PHASE II - FINAL REPORT

DEVELOPMENT OF BIOCATALYTIC REACTORS FOR REMOVAL OF VOLATILE CONTAMINANTS FROM PMMS, ECLSS AND LIFE SCIENCE WASTEWATER

VOLUME II

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APPENDIX A

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UNIBED SORBENT SIZING CALCULATIONS

OF THE ALCOHOL OXIDASE UNIBED

FOR THE

NASA-MSFC PHASE II SBIR - DEVELOPMENT OF

BIOCATALYTIC REACTORS FOR REMOVAL OF VOLATILE

CONTAMINANTS FROM PMMS, ECLSS AND LIFE SCIENCE WASTEWATER

Prepared By: _____

Approved By: _____ **Date:** _____

Approved By: _____ **Date:** _____

URC 80149

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1.0 INTRODUCTION

This document presents the design for the Alcohol Oxidase Unibed --a prototype Biocatalytic Reactor used for the removal of primary alcohols from waste water. Low molecular weight alcohols are not removed by adsorption. In this bed, they are oxidized at ambient temperature to adsorbable species (organic acids) that are subsequently removed by ion-exchange media. The adsorption media section of the unibed is configured for shorter life than the section containing catalyst material. Thus, breakthrough is detectable by an increase in conductivity. The alcohol oxidase unibed also imparts residual iodine into the effluent water, using iodinated resin.

This enzyme unibed should be installed downstream of a conventional unibed train in order to preclude or minimize interference from other waste water contaminants (see Figure 1). When it is paired with a urease unibed, it should follow that bed because urease is more robust than alcohol oxidase.

1.1 Applicable Document

1.1.1 SBIR Phase II Contract NAS8-38421

1.2 Applicable Drawings

1.2.1 Umpqua Research: URC DWG 90137

1.3 General Approach

The design is based on (1) laboratory data collected by small column testing on immobilized enzyme columns at UMPQUA between January 1988 and January 1991 under NASA contracts NAS8-37642 and NAS8-38421, and (2) isotherm data from shaker table and small column single contaminant, single media tests performed at UMPQUA under the following NASA contracts:

NAS9-17073	NAS9-17464
NAS9-17523	NAS9-17611

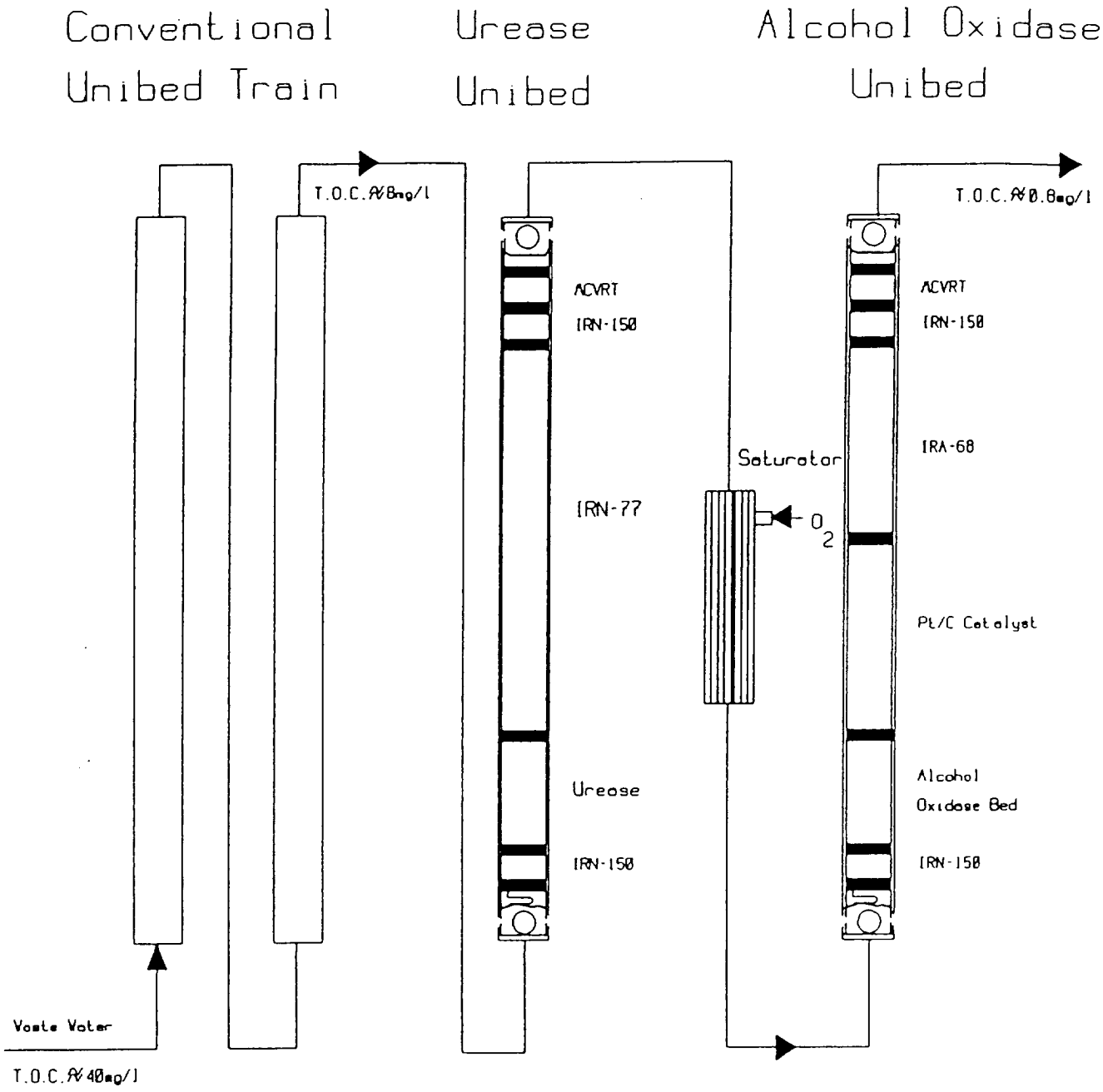


Figure 1. Location of Enzyme Unibeds

2.0 DESIGN REQUIREMENTS

2.1 Configuration

2.1.1 One 24 in long, 2 in diameter polycarbonate tube with an inlet and an outlet Quick Disconnect (QD).

2.2 Life at Design Conditions

2.2.1 Throughput: 1011 L

2.2.2 Time: 28 days

2.3 Inlet Solution

2.3.1 Potable Water Effluent from Potable Unibeds (URC #90086)

2.3.2 Ethanol: 16 mg/L

2.3.3 Methanol: 8 mg/L

2.4 Flow

2.4.1 Flow Rate: 25 mL/min ≈ 1.5 L/hr (3.3 lb/hr)

2.4.2 Daily Operating Time: 24 hr/day

2.4.3 1-Day Throughput: 36 L

2.5 Temperature

2.5.1 Operating Range: 68 - 77 F

2.6 Pressure

2.6.1 Maximum Operating Pressure (MOP): 40 psig

2.6.2 Proof Pressure: 60 ± 5 psig

2.7 Pressure Drop

2.7.1 Maximum Allowable Pressure Drop: 5 psig

2.8 Iodine Output

2.7.1 Range: 0.5 - 6.0 ppm

2.9 Outlet Quality

2.9.1 Water Quality Requirements: See Table 1. (NOTE: This standard applies prior to iodination.)

3.0 DESIGN DATA

The design data were developed by UMPQUA under contract to NASA-MSFC for the enzyme portion of the unibed and under contract to NASA-JSC for the ion exchange and MCV media (see paragraph 1.3 for applicable contract numbers).

3.1 Sorbent Selection

The best performing media have been selected for each bed, based on single adsorbent-single contaminant/shaker table and single adsorbent-single contaminant/dynamic column tests run previously by UMPQUA. The selected adsorbents are listed in Table 2.

3.2 Adsorption Equilibrium Data

Table 2 also contains ion exchange loadings (equilibrium data) necessary for the design of the sorption sub-beds. These data are from UMPQUA small-column tests and are lower than the manufacturer's published values.

4.0 UNIBED DESIGN

4.1 Unibed Dimensions

Each unibed consists of a single 2 in x 24 in long polycarbonate housing containing nominally, 1100 cc of media. The total bed length is 22 in. A sub-bed volume of 60 cc is the minimum bed length to diameter ratio necessary to insure proper sub-bed performance. The remaining volume is occupied by lip seals, an internal spring and the end caps.

TABLE 1. WATER QUALITY REQUIREMENTS
(Maximum Contaminant Levels)

QUALITY PARAMETERS	POTABLE WATER
PHYSICAL PARAMETERS	
Total Solids (mg/l)	100
Color, True (Pt/Co units)	15
Taste (TTN)	3
Odor (TON)	3
Particulates (max size - microns)	40
pH	6.0-8.5
Turbidity (NTU)	1
Dissolved gas (free @ 37 C)	Note 1
Free gas (@ STP)	Note 1
INORGANIC CONSTITUENTS (mg/l) (See Note 2)	
Ammonia	0.5
Arsenic	0.01
Barium	1.0
Cadmium	0.005
Calcium	30
Chloride	200
Chromium	0.05
Copper	1.0
Iodine (Total-includes organic iodine)	15
Iron	0.3
Lead	0.05
Magnesium	50
Manganese	0.05
Mercury	0.002
Nickel	0.05
Nitrate (NO_3-N)	10
Potassium	340
Selenium	0.01
Silver	0.05
Sulfate	250
Sulfide	0.05
Zinc	5.0

TABLE 1. WATER QUALITY REQUIREMENTS (Continued)
(Maximum Contaminant Levels)

QUALITY PARAMETERS	POTABLE WATER
ASTHETICS (mg/l)	
Cations	30
Anions	30
CO_2	15
MICROBIAL	
Bacteria (CFU/100 ml)	
Total Count	1
Anaerobes	1
Coliform	1
Virus (PFU/100 ml)	1
Yeast & Mold (CFU/100 ml)	1
RADIOACTIVE CONSTITUENTS (pCi/l)	Note 3
ORGANIC PARAMETERS (µg/l) (See Note 2)	
Total Acids	500
Cyanide	200
Halogenated Hydrocarbons	10
Phenols	1
Total Alcohols	500
Total Organic Carbon (TOC)	500
Uncharacterized TOC (UTOC)(See Note 4)	100
ORGANIC CONSTITUENTS (mg/l) (See Note 2)	

Note 1: No detectable gas using a volumetric gas vs fluid measurement system. Excludes CO_2 used for aesthetic purposes.
Note 2: Each parameter/constituent MCL must be considered individually and independently of others.
Note 3: The maximum contaminant levels for radioactive constituents in potable and personal hygiene water shall conform to Nuclear Regulatory Commission (NRC) regulations (10CFR20, et al.). These maximum contaminant levels are listed in the Federal Register, Vol. 51, No. 6, 1986, Appendix B, as Table 2 (Reference Level Concentrations) Column 2 (Water). Control/contaminant/monitoring of radioactive constituents used on SSF shall be the responsibility of the user. Prior to the introduction of any radioactive constituents on SSF, approval shall be obtained from the Radiation Constraints Panel (RCP). The RCP will approve or disapprove proposed monitoring and decontamination procedures on a case-by-case basis.
Note 4: UTOC equals TOC minus the sum of analyzed organic constituents expressed in equivalent TOC.

TABLE 2. SORPTION EQUILIBRIUM DESIGN VALUES

Contaminant	Media	Mfg Capacity meq/cc	URC Design Capacity mg/cc	Maximum Swelling
Acetic Acid	IRA-68	1.6	90	+ 20%
Formic Acid	IRA-68	1.6	101	+ 20%
Iodine (as I_2)	IRN-150	0.8	158	- 20%

TABLE 3. ALCOHOL OXIDASE UNIBED MEDIA CONFIGURATION

Flow Direction	Sorbent	Ref. Para	Volume (cc)	Life for EtOH=16 mg/L MeOH=8 mg/L (Days)	Function
↓	IRN-150	4.2.1	60	44	Iodine removal
	Alcohol Oxidase	4.2.2	200	56	Alcohol to aldehyde
	Platinum/Carbon Catalyst	4.2.3	360	56	Aldehyde to organic acid
	IRA-68	4.2.4	350	28	Remove organic acid
	IRN-150	4.2.5	60	44	Protect from I_2 backflow, scavenge aldehyde, organic acid
	MCV-RT	4.2.6	60	83	Microbial control
			1090 (1 tube)	28 days	

4.2 Unibed Configuration and Sub-bed Sizing

The configuration of the alcohol oxidase unibed is shown in Table 3. The initial IRN-150 sub-bed functions to remove iodine imparted into solution from a conventional unibed train. The biocatalyst alcohol oxidase sub-bed converts primary alcohols to their corresponding aldehydes. A platinum catalyst then oxidizes these aldehydes to organic acids. The IRA-68 sub-bed serves to remove this organic acid from the waste stream. A second sub-bed of IRN-150 resin provides additional polishing and protects the prior sub-beds from iodine in a reverse-flow situation. Finally, an MCV-RT resin sub-bed imparts iodine into the flow stream for microbial control. Since the waste water first passes through a conventional unibed assembly, the amount of ion-exchange media is sized solely for the organic acids, the major oxidation products of the catalyst sub-bed. The sizing rationale for each sub-bed is presented in the following paragraphs.

4.2.1 IRN-150

Waste water enters the alcohol oxidase unibed from a conventional unibed after passing through a urease unibed (see Figure 1). The water contains residual iodine, which impairs the activity of alcohol oxidase. Therefore, a 60 cc bed of IRN-150 is used to remove iodine. The minimum UMPQUA working capacity for IRN-150 is 158 mg I_2/cc.

Total Sorption Capacity: 60 cc IRN-150 x 158 mg I_2/cc = 9480 mg I_2

MCV^{RT} puts out 0.5 - 6 ppm I_2

Throughput Capacity: 9480 mg I_2 ÷ 6 mg I_2/L = 1580 L

Life: 1580 L ÷ 36 L/day = 44 days

4.2.2 Alcohol Oxidase.

Under laboratory conditions, a 14 cc bed of immobilized alcohol oxidase was tested with 20 ppmv ethanol at a flow rate of approximately 2.0 mL/min (empty bed contact time = 7 min). This bed converted over 95% of the ethanol to acetaldehyde, up to a throughput of 10 L/cc. The alcohol oxidase sub-bed was configured to have an empty bed contact time of 8 minutes. At the design flow of 25 mL/min, the sub-bed volume is 200 cc. For 20 ppmv ethanol, the throughput capacity of the enzyme sub-bed is considered to be 10 L/cc.

Life: 10 L/cc x 200 cc ÷ 36 L/day = 56 days.

4.2.3 Platinum/ Carbon Catalyst

Organic acids have a significantly higher sorption loading on IRA-68 than aldehydes do. Therefore, improved sorption efficiency is obtained by the oxidation of aldehydes to their corresponding organic acids. This is achieved by incorporating a catalyst bed consisting of platinum chemically reduced onto an activated carbon. Experimental data from small column tests with alcohol oxidase enzyme beds, done at UMPQUA, showed an optimum volume ratio of 2:1 catalyst bed to enzyme bed. Therefore, the size of the catalyst bed is

200 cc x 2 = 400 cc

The amount of platinum/carbon catalyst used in the unibed was lowered to 360 cc in order to accommodate an increase in the amount of resin used in the other sub-beds. The lifetime of this catalyst bed can be effected by the contaminant characteristics of the waste stream. If no inhibiting species reach this bed, its life should be at least as long as the alcohol oxidase sub-bed, i.e.: 56 days.

4.2.4 IRA-68

At low pH, acetic acid is adsorbed well by IRA-68, a weakly basic anion exchange resin, prepared by UMPQUA in the free base form. Using a compositional model based on 20 ppmv ethanol and 10 ppmv methanol, the amounts of acetic and formic acid formed in the alcohol oxidase sub-bed can be calculated as follows:

For 20 ppmv ethanol:

20 mL/1,000,000 mL x 0.79 g/mL x 1000 mL/L x 1000 mg/g = 15.8 mg/L

15.8 mg/L ÷ 46 mg/mmol ethanol = 0.35 mmol/L

0.35 mmol/L x 60 mg/mmol acetic acid = 21 mg/L acetic acid

For 10 ppmv methanol:

10 mL/1,000,000 x 0.79 g/mL x 1000 mL/L x 1000 mg/g = 7.9 mg/L

7.9 mg/L ÷ 32 mg/mmol methanol = 0.25 mmol/L

0.25 mmol/L x 46 mg/mmol formic acid = 11.5 mg/L formic acid

The UMPQUA working capacity for IRA-68 is 90 mg/cc for acetic acid and 101 mg/cc for formic acid. The volume of the IRA-68 portion of the bed in 350 cc.

Average capacity: (11.5 mg/L x 101 mg/cc + 21 mg/L x 90 mg/cc)

÷ (21 + 11.5) = 93.9 mg/cc

Total Sorption Capacity: 350 cc x 93.9 mg/cc = 32,865 mg

Throughput Capacity: 32,865 mg ÷ (11.5 + 21) mg/L = 1011 L

Life: 1011 L ÷ 36 L/day = 28 days

4.2.5 IRN-150

This 60 cc bed precedes the MCV^{RT} resin bed and serves to catch any iodine that may be emitted during a back flow situation. It also will scavenge traces of organic acid and unreacted aldehyde. The capacity of this sub-bed for iodine would be the same as calculated in 4.2.1. Its life is 44 days.

4.2.6 MCV^{RT} Resin

MCV^{RT} resin is required at the exit of each unibed for microbial control. The resin puts out 0.5 - 6 ppm I_2 for a duration of 50 L/cc media. The lifetime of this sub-bed is:

Life: 60 cc x 50 L/cc ÷ 36 L/day = 83 days

4.2.7 Sizing Discussion

The design summarized in Table 3 was obtained within the dimension restraints given in Paragraph 4.1. Unibed life could be doubled from 28 days to 56 days by doubling the sizes of the IRA-68 sub-bed from 350 cc to 700 cc. The total amount of media would increase from 1090 cc to 1440 cc. Thus a 100% increase in life would result from a 32% increase in bed size. A further improvement would result from adjusting the size of each sub-bed to match the 83 day life of the MCV-RT resin.

4.3 Pressure Drop

Previous testing developed a pressure drop equation.

$\delta P = 0.4\ WL\ \mu/D^2$

where:

δP = Pressure drop, psi

W = flow rate, lb/min

L = bed length, in

D = bed diameter, in

μ = viscosity, centipoise

For the enzyme unibeds:

W = 2.5 lb/hr = 0.0417 lb/min

L = 21.5 in

D = 2 in

μ = 1 centipoise

$\delta P = 0.4\ (0.00417)(21.5)(1)/(2)^2 = 0.9$ psi

Specified max δP = 5.0 psi

4.4 Summary of Unibed Design Values

A summary of the design values for the enzyme beds is given in Table 4.

TABLE 4. SUMMARY OF ALCOHOL OXIDASE UNIBED DESIGN VALUES

Parameter		Value
URC Drawing Number		90137
Nominal ID		2 in
Water System		Potable
Flow Rate		3.3 lb/hr (1.5 L/hr)
Daily Operating Time		24 hr/day
Thruput, 1 day		36 L
Total Media Volume		1090 cc
Cross Sectional Area		20.3 cm^2
Total Length of Media (Installed)		22 in
Face Velocity		1.23 cm/min
Empty Bed Contact Time	- Enzyme Subbed	8 min
	- Catalyst Sub-bed	14 min
	- Unibed	44 min
Life (limited by IRA-68)	-	1011 L
	-	28 days

APPENDIX I

MEDIA INFORMATION

IRN-150

IRN-68

ROHM AND HAAS COMPANY
PHILADELPHIA, PENNSYLVANIA 19105
FLUID PROCESS CHEMICALS

AMBERLITE ION EXCHANGE RESINS

AMBERLITE® IRN-150

Amberlite IRN-150 is a mixture of gelular, polystyrene cation and anion exchange resins. Amberlite IRN-150 resin as supplied contains a stoichiometric equivalent of the strongly acidic cation (Amberlite IRN-77) and the strongly basic anion (Amberlite IRN-78) exchange resins. It is supplied in the hydrogen/hydroxide form as clear, amber colored spherical particles virtually perfect in bead appearance. Amberlite IRN-150 resin is designed for use in industrial water treatment applications, particularly in once through applications such as primary water chemistry control in nuclear power operations. This resin combines the properties of high capacity and excellent resistance to bead fracture from attrition or osmotic shock.

Amberlite IRN-150 resin is designated as a Nuclear Grade resin and is manufactured using special processing procedures. These procedures, combined with a patented Rohm and Haas process to reduce the chloride content of the anion component, produce material of the ultimate purity and yield a product meeting the exacting demands of the nuclear industry. Amberlite IRN-150 resin is recommended in any non-regenerable mixed bed application where reliable production of the highest quality water is required and where the "as supplied" resin must have an absolute minimum of ionic and non-ionic contamination.

IMPORTANT FEATURES OF AMBERLITE IRN-150 ION EXCHANGE RESIN

HIGH CAPACITY: Amberlite IRN-150 resin will exhibit a nominal operating capacity of 12 kg/ft³ (0.55 meq/ml).

EXCEPTIONAL PURITY: Amberlite IRN-150 resin is manufactured to demanding purity specifications which assure a minimum of ionic and non-ionic contamination.

GOOD RESISTANCE TO BEAD FRACTURE: Amberlite IRN-150 resin offers superior performance with respect to particle breakdown from attrition or osmotic shock.

INSOLUBLE IN ALL COMMON SOLVENTS

RECOMMENDED CONDITIONS OF OPERATION

The recommended conditions for operation of Amberlite IRN-150 resin are listed below.

BED DEPTH: 24" minimum (0.61 m)

SERVICE FLOW RATE: 2–5 gpm/ft³ (16 to 40.1 l/hr/l)

PHYSICAL CHARACTERISTICS

SHAPE: Spherical beads

SHIPPING WEIGHT: 43 lbs/ft³ (688 g/l)

PARTICLE SIZE (U.S. MESH):

Screen Size	% Maximum
+16	5.0
−40	5.0
−50	0.5

PERFECT BEADS: 95% minimum

CHEMICAL CHARACTERISTICS

IONIC FORM: Hydrogen/Hydroxide

CATION TO ANION EQUIVALENT RATIO: 1:1

Ionic Content by Individual Component:	IRN-77	IRN-78
Equivalent % H, minimum	99.0	na
Equivalent % OH, minimum	na	95.0
Equivalent % Cl, maximum	na	0.10
Equivalent % CO_3, maximum	na	5.0
Equivalent % SO_4, maximum	na	0.10
Sodium (ppm dry resin) maximum	50	50
Iron (ppm dry resin) maximum	50	50
Copper (ppm dry resin) maximum	10	10
Heavy metals as Pb (ppm dry resin) maximum	10	10
Aluminum (ppm dry resin) maximum	50	50
Calcium (ppm dry resin) maximum	50	50
Magnesium (ppm dry resin) maximum	50	50

OHM AND HAAS COMPANY
PHILADELPHIA, PENNSYLVANIA 19105
FLUID PROCESS CHEMICALS

AMBERLITE ION EXCHANGE RESINS

AMBERLITE® IRA-68

Amberlite IRA-68 is a gel type, weakly basic anion exchange resin possessing tertiary amine functionality in a crosslinked acrylic matrix. In addition to exhibiting a high exchange capacity, this resin has good chemical and thermal stability and is especially suited to the adsorption and desorption of organic materials from solution. Amberlite IRA-68 is also well suited for applications in the pharmaceutical, chemical and food processing industries for the neutralization of strong acids and other special processes.

IMPORTANT FEATURES OF AMBERLITE IRA-68

HIGH CAPACITY AND LOW COST REGENERATION — Amberlite IRA-68 has an operating acid removal exchange capacity of 29 kgrs/ft³ (66.4 g/l as $CaCO_3$) of resin. Regeneration is accomplished using 110-120% of the quantity of base chemically equivalent to the operating capacity. Thus, regenerant costs are significantly lower than for strongly basic resins and waste problems are held at a minimum.

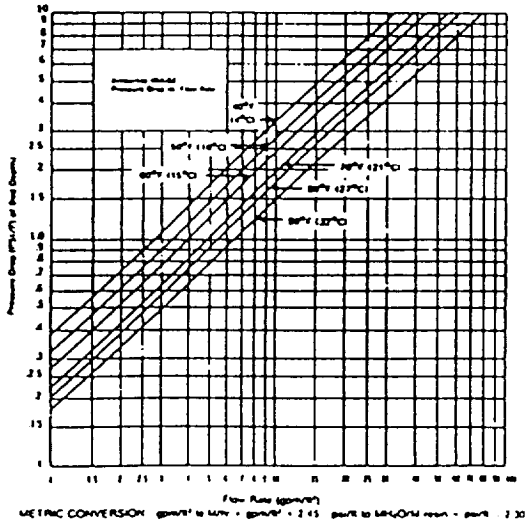
RESISTANCE TO ORGANIC FOULING — Amberlite IRA-68 is synthesized with an open structure which permits the effective adsorption and desorption of large organic molecules. Because of this open structure, organic materials are readily eluted from Amberlite IRA-68 resulting in no capacity loss due to organic fouling.

CHEMICAL FORM — Amberlite IRA-68 is shipped in the fully regenerated free-base form and can be utilized immediately for acid removal.

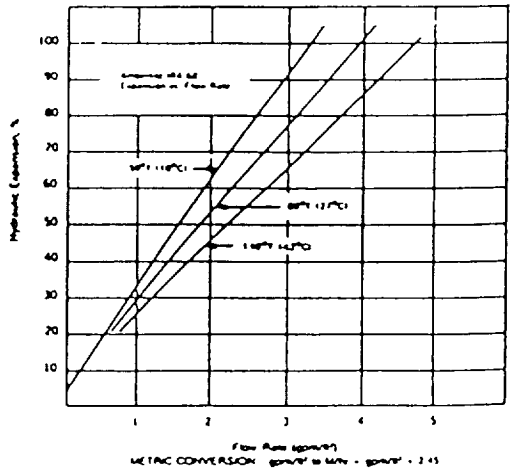
INSOLUBLE IN ALL COMMON SOLVENTS.

HYDRAULIC CHARACTERISTICS

PRESSURE DROP — The curves show the expected pressure drop per foot of bed depth in normal downflow operation at various temperatures as a function of flow rate.



BACKWASH CHARACTERISTICS — After each operational cycle Amberlite IRA-68 should be backwashed for approximately ten minutes to re-classify the resin particles and purge the bed of any insoluble material which may have collected on top of the resin. The resin bed should be expanded a minimum to 50% during backwash.



PHYSICAL CHARACTERISTICS

PHYSICAL FORM — Uniform, spherical particles shipped in moist, fully regenerated condition.

DENSITY — 41 to 47 lbs/ft³ (656 to 752 g/l)

SHIPPING WEIGHT — 45 lbs/ft³ (720 g/l)

MOISTURE CONTENT — 60% as shipped *

SCREEN GRADING (WET) — 16 to 50 mesh (U.S. Standard Screen)

EFFECTIVE SIZE — 0.45 mm.*

FINES CONTENT — 3% maximum

SWELLING — 20%* upon complete conversion of the resin from the free base to the chloride form.

*Approximate

SUGGESTED OPERATING CONDITIONS

Limitation — 0 to 7
Maximum Temperature — 140°F (60°C)
Minimum Bed Depth — 24 inches (0.61 m)
Backwash Flow Rate — See detailed information
Regenerant Concentration* — 4%
Regenerant Flow Rate — 0.5 to 1.0 gpm/ft³ (4.0 to 8.0 l/hr/l)
Regeneration Level — See detailed information
Rinse Flow Rate — 0.5 gpm/ft³ (4.0 l/hr/l) initially, to displace regenerant then 1.5 gpm/ft³ (12.0 l/hr/l)
Rinse Water Requirements — 50 to 75 gal/ft³ (6.7 to 10.1 l/l)
Service Flow Rate — 1 to 3 gpm/ft³ (8.0 to 24.1 l/hr/l)
Exchange Capacity — See detailed information
*See Safe Handling Information section

REGENERATION LEVEL AND CAPACITY

A minimum acid removal operating capacity of 28 kgrs. (as $CaCo_3/ft^3$) (64 g/l) of resin may be expected using the following amounts of regenerants:
3.7 lbs of NaOH/ft³ (59.2 g/l) or
3.2 lbs of NH_4OH/ft³ (51.2 g/l) or
4.9 lbs of Na_2Co_3/ft³ (78.4 g/l)

APPLICATIONS

DEIONIZATION — The marked worldwide increase in the use of acrylic anion exchange resins is illustrated by the increased utilization of Amberlite IRA-458 as the strongly basic anion exchange component of many deionization systems. Amberlite IRA-458 is installed when high capacity, excellent organic fouling resistance, and good physical stability are required.

Where plant design, however, dictates the use of a weakly basic anion exchange resin with properties comparable to those of Amberlite IRA-458, Amberlite IRA-68 is the prime choice. Amberlite IRA-68 is a gelular acryic weakly basic anion exchange resin with tertiary amine functionality. The acrylic matrix of Amberlite IRA-68 is hydrophilic making it similar to that of Amberlite IRA-458. When compared with gelular polystyrene or epoxy-amine type resins, the acrylic matrix of Amberlite IRA-68 shows superior kinetic behavior particularly in regeneration elution of organics. This superior organic fouling resistance places Amberlite IRA-68 in the same class as *macroreticular* styrene weakly base anion exchange resins.

The flexible nature of the gelular acrylic matrix imparts excellent physical stability with regard to mechanical attrition, and osmotic shock. This, again, is normally attributed to a macroreticular structure.

In contrast to most weakly basic anion exchange resins, the working capacity of Amberlite IRA-68 is independent of service flow rate (1.0 to 5.0 gpm/ft³ [8.0 to 40.1 l/hr/l]), temperature (40°–70°F [4 to 21°C]), and only slightly affected by influent water analysis changes. A base working capacity of 29.0 kgr/ft³ (64 g/l) can be expected under normal operating conditions.

The weakly basic anion exchange resin Amberlite IRA-68 incorporates the high working capacity of gel styrene and gel epoxy-amine weakly basic anion exchange resins, without the latter resins' inherent physical weaknesses and organic fouling tendencies. At the same time, it also incorporates the superior physical stability and organic fouling resistance associated with macroreticular weakly basic anion exchange resins, while avoiding the lower working capacities normally associated with macroreticular structure.

ACID MINE DRAINAGE — A modification of the DESAL Process for the treatment of acid mine drainage water has been developed in the Rohm and Haas laboratories. This process, utilizing Amberlite IRA-68 operating in the bicarbonate cycle, converts metallic sulfates, the principal anionic constituents of AMD waters, into soluble bicarbonates which when aerated precipitate as insoluble hydrous oxides. The resulting effluent water will contain calcium and magnesium hardness, which if desired, can be softened using a cold lime softening treatment.

DEIONIZATION AND ORGANIC SCAVENGING — Amberlite IRA-68 is particularly suited for the removal of strong acids and the deionization of process liquors. This resin should be considered for use in the deionization of water and special applications where high molecular weight materials are to be removed from solution.

DEASHING AND DECOLORING CORN SUGAR — When properly pretreated Amberlite IRA-68 is cleared for use in food processing under FDA Food Additive Regulation 21CFR-173.25. According to this regulation the food or aqueous flow must be maintained at 50°C or below, and the flow through the resin must be less than 0.5 gpm/ft³ (4.0 l/hr/l).

SAFE HANDLING INFORMATION — A Material Safety Data Sheet is available for Amberlite IRA-68. To obtain a copy contact your Rohm and Haas representative.

Caution: Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact.

Nitric acid and other strong oxidizing agents can cause explosive type reactions when mixed with ion exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidizing agent such as nitric acid is contemplated. Before using strong oxidizing agents in contact with ion exchange resins, consult sources knowledgeable in the handling of these materials.

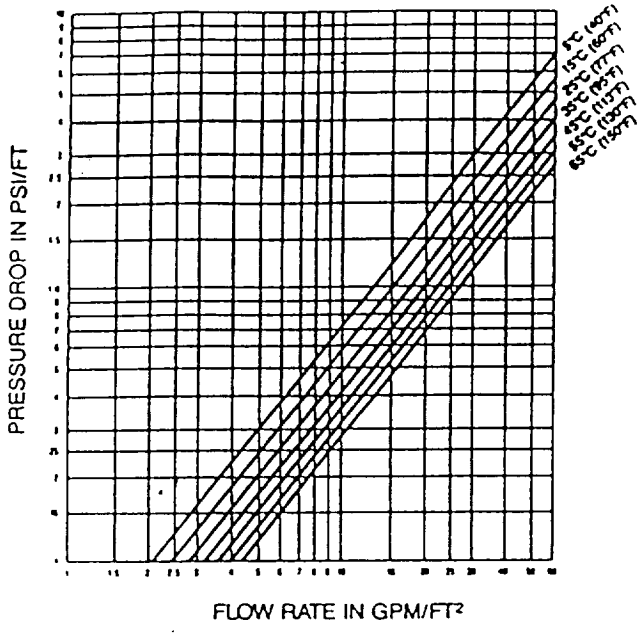
IE-120-67/80 June 1982 Printed in U.S.A.

HYDRAULIC CHARACTERISTICS

PRESSURE DROP: The approximate pressure drop for each foot of bed depth of Amberlite IRN-150 resin in normal down flow operation at various temperatures and flow rates is shown in the graph below.

RESIN HANDLING: To retain the high purity standards of nuclear grade resins, deionized water should be used for all resin handling. Contact of the resin with air should also be minimized to avoid CO_2 pickup and subsequent loss of capacity of the anion resin.

AMBERLITE® IRN-150 RESIN
PRESSURE DROP



METRIC CONVERSION GPM/ft² to M hr = GPM/ft² x 2.45
PSI/ft to MH₂O/M resin = PSI/ft x 2.30

APPLICATIONS

MIXED BED DEIONIZATION: The physical and chemical characteristics of Amberlite IRN-150 resin provide excellent performance when used in production of high quality water in any mixed bed deionization application.

NUCLEAR APPLICATIONS: The purity and physical stability of Amberlite IRN-150 resin provides unsurpassed performance in nuclear applications such as chemistry control in primary water treatment. Amberlite IRN-150 resin can also be used for a variety of rad waste applications.

PRODUCTION OF ULTRA PURE WATER: Amberlite IRN-150 resin is an excellent choice for once through (non-regenerable) applications typically found in the final DI water processing for the semiconductor industry. Amberlite IRN-150 resin provides rapid rinse to 18 megohm, high capacity, and reliable production of the highest-quality water.

SAFE HANDLING INFORMATION

A Material Safety Data Sheet is available for Amberlite IRN-150 resin. To obtain a copy, contact your Rohm and Haas representative.

CAUTION: Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact.

Nitric acid and other strong oxidizing agents can cause explosive type reactions when mixed with ion exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidizing agent such as nitric acid is contemplated. Before using strong oxidizing agents in contact with ion exchange resins, consult sources knowledgeable in the handling of these materials.

APPENDIX II

MATERIAL SAFETY DATA SHEETS

IRN-150

ALCOHOL OXIDASE BED

PLATINUM CATALYST ON CARBON

IRA-68

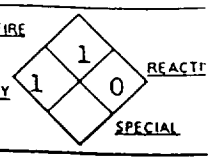
MCV-RT IODINATED RESIN

ROHM AND HAAS COMPANY

CORPORATE PRODUCT INTEGRITY DEPARTMENT
INDEPENDENCE MALL WEST
PHILADELPHIA, PA 19105

EMERGENCY TELEPHONE
215-592-3000 (ROHM AND HAAS)
800-424-9300 (CHEMTREC)

HAZARD RATING
4=EXTREME
3=HIGH
2=MODERATE
1=SLIGHT
0=INSIGNIFICANT
*=SEE SECTION IV



BS242
LIST 7

MATERIAL SAFETY DATA SHEET

NOT OSHA HAZARDOUS
NOT WHMIS CONTROLLED

MATERIAL	CODE	KEY	DOT HAZARD CLASS
AMBERLITE® IRN-150 Resin	69855	891090-3	NON-REGULATED
	DATE ISSUED 11/08/88		

FORMULA	CHEMICAL NAME OR SYNONYMS
Not applicable	Mixed bed ion exchange resin (hydrogen and hydroxide forms)

I – COMPOSITIONAL INFORMATION

	CAS Reg. No.	APPROX WT %	TWA/TLV
			R&H OSHA ACGIH
Anion/cation exchange resin	NONHAZ	35-50	NE NE NE
Water	NONHAZ	50-65	NE NE NE
			NE = None established

II – PHYSICAL PROPERTY INFORMATION

APPEARANCE - ODOR - pH.			VISCOSITY
Beads; pH (aqueous slurry) = 5 to 9			NA
MELTING OR FREEZING POINT	BOILING POINT	VAPOR PRESSURE (mm Hg)	VAPOR DENSITY (AIR=1)
0C/32F (water)	100C/212F (water)	17 @20C (water)	Less than 1 (water)
SOLUBILITY IN WATER	PERCENT VOLATILE (BY WEIGHT)	SPECIFIC GRAVITY (WATER=1)	EVAPORATION RATE (BUTYL ACETATE=1)
Negligible	50-65 (water)	1.1-1.3	Less than 1 (water)

III – FIRE AND EXPLOSION HAZARD INFORMATION

FLASH POINT	AUTO IGNITION TEMPERATURE	LOWER EXPLOSION LIMIT (%)	UPPER EXPLOSION LIMIT (%)
NA	500C/932F (est.)	NA	NA

EXTINGUISHING MEDIA

☐ FOAM ☐ "ALCOHOL" FOAM ☒ CO_2 ☒ DRY CHEMICAL ☒ WATER SPRAY ☐ OTHER

SPECIAL FIRE FIGHTING PROCEDURES

Wear self-contained breathing apparatus (pressure-demand, MSHA/NIOSH-approved or equivalent) and full protective gear.

UNUSUAL FIRE AND EXPLOSION HAZARDS

Toxic combustion products may include alkylamines and oxides of sulfur and nitrogen.

IV – HEALTH HAZARD INFORMATION

ROHM AND HAAS RECOMMENDED WORK PLACE EXPOSURE LIMITS

STEL = None established.

EFFECTS OF OVEREXPOSURE

Eye Contact: Product can cause eye irritation.

EMERGENCY AND FIRST AID PROCEDURES

Eye Contact: Immediately flush eyes with large amounts of water and continue for at least 15 minutes. Get prompt medical attention.

FORM 898 REV. 11/88

V – REACTIVITY INFORMATION

STABILITY: [X] STABLE [ ] UNSTABLE

CONDITIONS TO AVOID: Temperatures over 200C/392F.

HAZARDOUS DECOMPOSITION PRODUCTS: Thermal decomposition may yield styrene monomer, divinylbenzene, alkylamines and oxides of sulfur and nitrogen.

HAZARDOUS POLYMERIZATION: [ ] MAY OCCUR [X] WILL NOT OCCUR

CONDITIONS TO AVOID: None known

INCOMPATIBILITY (MATERIALS TO AVOID): [ ] WATER [X] OTHER

Avoid contact with concentrated nitric acid or any other strong oxidizing agent at all times.

VI – SPILL OR LEAK PROCEDURE INFORMATION

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED

Floor may be slippery. Use care to avoid falls. Sweep up and transfer to containers for recovery or disposal.

WASTE DISPOSAL METHODS: Unused resin may be incinerated or landfilled in facilities meeting local, state and federal regulations. For contaminated resin, the user must determine the hazard and use an appropriate disposal method.

VII – SPECIAL PROTECTION INFORMATION

VENTILATION TYPE: Normal room ventilation.

RESPIRATORY PROTECTION: None required for normal operations.

PROTECTIVE GLOVES	EYE PROTECTION
None required	Safety glasses (ANSI Z-87.1 or approved equivalent)

OTHER PROTECTIVE EQUIPMENT: Eyewash facility

VIII – STORAGE AND HANDLING INFORMATION

STORAGE TEMPERATURE	INDOOR	HEATED	REFRIGERATED	OUTDOOR
MAX. 49C/120F MIN. 0C/32F	YES	NO	NO	YES

NOTE: Store at ambient temperatures. Avoid repeated freeze-thaw cycles.

NOTE: Ground ion exchange resins should be treated as potential eye irritants. A finely ground form of a structurally related strong acid cation exchange resin produced severe rabbit eye irritation.

NOTE: The maximum operating temperature for this product is 60C/140F. Functional group destruction and loss of capacity will occur above this temperature.

IX – TOXICITY INFORMATION

No toxicity data available for this product.

X – MISCELLANEOUS INFORMATION

Caution: Do not pack column with dry ion exchange resins. Dry beads expand when wetted; this expansion can cause a glass column to shatter.

Caution: Nitric acid and other strong oxidizing agents can cause explosive-type reactions when mixed with ion exchange resins. Proper design of equipment to prevent rapid build-up of pressure is necessary if use of an oxidizing agent such as nitric acid is contemplated. Before using strong oxidizing agents in contact with ion exchange beads, consult sources knowledgeable in handling these materials.

NA • NOT APPLICABLE C • CEILING VALUE	KEY	DATE OF ISSUE	SUPERSEDES
	891090-3	11/08/88	09/04/87

MATERIAL SAFETY DATA SHEET

Umpqua Research Company
P.O. Box 791 - 125 Volunteer Way
Myrtle Creek, OR 97457
(503) 863-7770

Feb. 25, 1991

----------IDENTIFICATION----------

PRODUCT #: 90021-57 NAME: Alcohol Oxidase Bed

----------HAZARDOUS INGREDIENTS----------

Alcohol Oxidase enzyme on a silica base which has platinum deposited on the surface.

----------PHYSICAL/CHEMICAL CHARACTERISTICS----------

Grey-orange powder or granules; free flowing when dry.

----------FIRE AND EXPLOSION HAZARD DATA----------

Avoid open flames. May emit toxic fumes under fire conditions. Wear self-contained breathing apparatus. The silica base is non-flammable. Enzyme fire may be extinguished using water, foam, dry chemical or CO_2 extinguisher.

----------REACTIVITY DATA----------

Avoid close proximity to volatile solvents. Do not contact with reducing agents or strong oxidizing agents.

----------HEALTH HAZARD DATA----------

This product contains crystalline silica, which is considered a hazard by inhalaton. IARC has classified crystalline silica as a probable carcinogen for humans, although NTP and OSHA have not. Silica is also a know cause of silicosis, a non-cancerous lung disease, when inhaled repeatedly at high levels.

The alcohol oxidase enzyme may be harmful if ingested, and the polypeptide material may cause allergic sensitization upon repeated skin contact.

Platinum compounds are pulmonary sensitizers, and may cause skin irritation.

----------PRECAUTIONS FOR SAFE HANDLING AND USE----------

Store away from volatile organic solvents. Store wet, under water. Do not ingest or inhale. Avoid eye contact.

----------CONTROL MEASURES----------

Use rubber gloves and wear goggles when handling. If spilled, clean up with broom and dustpan. This material may be land-filled as ordinary trash. Avoid raising dust.

URC 80147

MATERIAL SAFETY DATA SHEET

Umpqua Research Company
P.O. Box 791 - 125 Volunteer Way
Myrtle Creek, OR 97457
(503) 863-7770

Feb. 20, 1991

----------IDENTIFICATION----------

PRODUCT #: 90021-59 NAME: Platinum Catalyst on Carbon

----------HAZARDOUS INGREDIENTS----------

The activated carbon base material is non-hazardous per Office of Hazardous Materials Management. Platinum compounds, which are present only in small quantities, are pulmonary sensitizers. Platinum compounds may be harmful by inhalation, ingestion or skin absorption.

----------PHYSICAL DATA----------

Appearance: Black irregular granules; free flowing when dry; no odor.

----------FIRE AND EXPLOSION HAZARD DATA----------

Avoid open flames. May emit toxic fumes under fire conditions. Wear self-contained breathing apparatus. Use water, foam, dry chemical or carbon dioxide fire extinguisher.

----------REACTIVITY DATA----------

Avoid close proximity to volatile solvents, strong oxidizing or reducing chemicals.

----------HEALTH HAZARD DATA----------

Inhalation:

Acute Exposure: No effects reported for platinum metal. Some platinum compounds cause coughing, wheezing, sneezing, bronchitis and dyspnea.

Chronic Exposure: No effects reported for the metal. Some individuals may develop allergic reactions to some platinum salts or solutions, which may cause asthma and bronchitis.

First Aid: Remove from exposure. If breathing has stopped, perform artificial respiration. If breathing is difficult, give oxygen. Keep patient warm and at rest. Get medical attention.

Skin Contact:

Acute Exposure: No effects reported for the metal. Some platinum compounds may cause urticaria, a skin disease.

Chronic Exposure: No effects for the metal. Some individuals may develop allergic reactions to some platinum compounds which may cause sensitization dermatitis.

First Aid: Remove contaminated clothing and shoes immediately. Wash affected area with soap or mild detergent and large amounts of water until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

MATERIAL SAFETY DATA SHEET

Umpqua Research Company
P.O. Box 791 - 125 Volunteer Way
Myrtle Creek, OR 97457
(503) 863-7770

Feb. 20, 1991

----------IDENTIFICATION----------

PRODUCT #: 90021-59 NAME: Platinum Catalyst on Carbon

----------HEALTH HAZARD DATA----------

Continued

Eye Contact:

Acute Exposure: No effects reported for the metal. Some platinum compounds cause irritation and lacrimation.

Chronic Exposure: No effects reported for the metal. Some platinum compounds cause conjunctivitis.

First Aid: Wash eyes immediately with large amounts of water, occasionally lifting upper and lower lids, until no evidence of chemical remains (approximately 15-20 minutes). Get medical attention immediately.

Ingestion:

Acute Exposure: No applicable information was found.

Chronic Exposure: No applicable information was found.

First Aid: Do not induce vomiting in unconscious victim. Get medical attention immediately.

Toxicology:

No applicable information found for platinum metal. Some platinum compounds are pulmonary sensitizers. Soluble platinum salts are primary skin irritants and skin sensitizers.

----------PRECAUTIONS FOR SAFE HANDLING AND USE----------

Store away from volatile organic solvents. Damp carbon absorbs O_2 gas from the atmosphere. Use self-contained breathing apparatus when dealing with large quantities. May be stored completely dry or in water.

----------CONTROL MEASURES----------

Use dust or particle mask, rubber gloves, and goggles. If spilled, clean up with broom and dustpan. Umpqua Research Company will accept return of spent catalyst. Material may be disposed of in a landfill.

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. UMPQUA RESEARCH COMPANY SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT.

URC 80146

SIGMA® chemical company

THE WORLD'S FOREMOST MANUFACTURER OF RESEARCH BIOCHEMICALS AND DIAGNOSTIC REAGENTS

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CONTINENTAL U.S.A (EXCL MO)
1-800-325-8070
OR PHONE COLLECT
1-314-771-5765
FROM ANYWHERE IN THE WORLD
MAILING ADDRESS: P.O. BOX 14508, ST. LOUIS, MO. 63178, U.S.A.

CABLE ADDRESS: SIGMACHEM TWX: 910-761-0593

ATTN: SAFETY DIRECTOR
UMPQUA RESEARCH COMPANY
P O BOX 791
MYRTLE CREEK OR 97457

EMERGENCY PHONE 1-314-771-5765

DATE: 07/22/86

CUST#: 4-073-87920 PO#: 245

MATERIAL SAFETY DATA SHEET

IDENTIFICATION

STOCK #: IRA-68
PRODUCT #: A7018
CAS #: 9056-59-1

NAME: AMBERLITE RESIN FREE BASE FORM GEL TYPE

TOXICITY HAZARDS

DATA NOT AVAILABLE

HEALTH HAZARD DATA

ACUTE EFFECTS
MAY CAUSE EYE IRRITATION.
DUST OR PARTICLES MAY IRRITATE THE EYES AS ANY FOREIGN BODY.

FIRST AID
IF SWALLOWED, WASH OUT MOUTH WITH WATER. CALL A PHYSICIAN.
IN CASE OF SKIN CONTACT, FLUSH WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES. REMOVE CONTAMINATED CLOTHING AND SHOES AND CALL A PHYSICIAN.
IF INHALED, REMOVE TO FRESH AIR. IF BREATHING BECOMES DIFFICULT, CALL A PHYSICIAN.
IN CASE OF CONTACT WITH EYES, FLUSH WITH COPIOUS AMOUNTS OF WATER FOR AT LEAST 15 MINUTES. ASSURE ADEQUATE FLUSHING BY SEPARATING THE EYELIDS WITH FINGERS. CALL A PHYSICIAN.

PHYSICAL DATA

SPECIFIC GRAVITY: 1.06
SOLUBILITY: WATER-INSOLUBLE

APPEARANCE AND ODOR
OFF-WHITE BEADS, SLIGHT AMINE ODOR.

FIRE AND EXPLOSION HAZARD DATA

AUTOIGNITION TEMPERATURE: 427°C

EXTINGUISHING MEDIA
CARBON DIOXIDE.
DRY CHEMICAL POWDER.
WATER SPRAY.

SPECIAL FIREFIGHTING PROCEDURES
WEAR SELF-CONTAINED BREATHING APPARATUS AND PROTECTIVE CLOTHING TO PREVENT CONTACT WITH SKIN AND EYES.

REACTIVITY DATA

STABILITY
STABLE.

CONDITIONS TO AVOID
TEMPERATURES ABOVE 220°C

INCOMPATIBILITIES
NITRIC ACID AND OTHER STRONG OXIDIZING AGENTS CAN FORM EXPLOSIVE TYPE REACTIONS WHEN MIXED WITH ION EXCHANGE RESINS.

HAZARDOUS COMBUSTION OR DECOMPOSITION PRODUCTS
ACRYLIC MONOMER, DIVINYLBENZENE

CONTINUED ON NEXT PAGE

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CABLE ADDRESS: SIGMACHEM TWX: 910-761-0593

STOCK #: IRA-68 CUST#: 4-073-87920 PO#: 245

PRODUCT #: A7018 NAME : AMBERLITE RESIN FREE BASE FORM GEL TYPE

---------- REACTIVITY DATA ----------

TOXIC FUMES OF:
CARBON MONOXIDE AND CARBON DIOXIDE
NITROGEN OXIDES
ZARDOUS POLYMERIZATION
WILL NOT OCCUR.

---------- SPILL OR LEAK PROCEDURES ----------

EPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED
WEAR RESPIRATOR, CHEMICAL SAFETY GOGGLES, RUBBER BOOTS AND HEAVY RUBBER GLOVES.
SWEEP UP, PLACE IN A BAG AND HOLD FOR WASTE DISPOSAL.
FLOOR MAY BE SLIPPERY
VENTILATE AREA AND WASH SPILL SITE AFTER MATERIAL PICKUP IS COMPLETE.
ASTE DISPOSAL METHOD
THIS MATERIAL MAY BE LANDFILLED AS ORDINARY TRASH.
OBSERVE ALL FEDERAL, STATE, AND LOCAL LAWS.

--- PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE ---

OSHA/MSHA-APPROVED RESPIRATOR.
MECHANICAL EXHAUST.
COMPATIBLE CHEMICAL RESISTANT GLOVES.
CHEMICAL SAFETY GOGGLES.
ORY ION EXCHANGE RESINS EXPAND WHEN WETTED, WHICH MAY CAUSE COLUMN TO SHATTER.

HE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE L INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. SIGMA SHALL NOT BE HELD ABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE OVE PRODUCT. SEE REVERSE SIDE OF INVOICE OR PACKING SLIP FOR ADDITIONAL ERMS AND CONDITIONS OF SALE.

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DORSET BH17 7NH
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A M BAHNSTEIG 7
D-8012 TAUFKIRCHEN
WEST GERMANY

UMPQUA RESEARCH COMPANY
P.O. BOX 791 - 626 N.E. DIVISION
MYRTLE CREEK, OREGON 97457
(503) 863-5201
FAX (503) 863-6199

MATERIAL SAFETY DATA SHEET

Feb. 20, 1991

----------IDENTIFICATION----------

PRODUCT #: 90021-47 NAME: MCV-RT Iodinated Resin

----------TOXICITY HAZARDS----------

Effects of Overexposure: Can irritate eyes, nose, throat and skin, hypersensitivity, nausea, abdominal pain, diarrhea, excessive thirst, circulatory failure. Possibly fatal if swallowed.

----------HEALTH HAZARD DATA----------

Threshold Limit Value
TVL-air: 0.1 ppm as Iodine TXDS: orl-Hmn LDLo: 5 mg/kg as Iodine
First Aid Procedures:
Skin: wash with soap/water; get medical assistance.
Eyes: flush thoroughly with water 15 minutes. Assure adequate flushing by separating the eyelids with fingers; get medical assistance.
Inhalation: remove to fresh air; get medical assistance.
Ingestion: give milk, starch solution, or tablespoon sodium thiosulfate in a glass of water and get immediate medical attention. Treat for shock.
Acute Effects: may cause eye irritation. Particles can irritate the eyes. Finely ground particles of similar material caused corneal damage in rabbit eyes.

----------PHYSICAL DATA----------

Specific Gravity: 1.11
Appearance and Odor: Dark purple to black beads, with moderate iodine and amine odor.
Solubility: Beads release iodine in water in concentrations below 300 ppm

----------FIRE AND EXPLOSION HAZARD DATA----------

Autoignition Temperature: 427 C EST
Extinguishing Media
Carbon Dioxide
Dry Chemical Powder
Water Spray
Special Firefighting Procedures
Wear Self-contained breathing apparatus and protective clothing to prevent contact with skin and eyes.
Unusual Fire and Explosions hazards
Emits Toxic fumes under fire conditions.

URC 80145

UMPQUA RESEARCH COMPANY

P.O. BOX 791 - 626 N.E. DIVISION
MYRTLE CREEK, OREGON 97457
(503) 863-5201
FAX (503) 863-6199

MATERIAL SAFETY DATA SHEET

Feb. 20, 1991

--------IDENTIFICATION--------

PRODUCT #: 90021-47 **NAME:** MCV-RT Iodinated Resin

----------REACTIVITY DATA----------

Drying results in release of iodine vapor.
Stability: stable.
Conditions to avoid: Temperatures over 220 C.

Incompatibilities: Nitric Acid and other strong Oxidizing agents can cause explosion.
Materials to avoid: NH_3, Acetylene, Acetaldehyde, Active metals particularly powdered Al.
Reactions when mixed with ion exchange resins.
Hazardous combustion or decomposition products.
Styrene Monomer, Divinylbenzene
Toxic fumes of:
Carbon Monoxide and Carbon Dioxide
Nitrogen Oxides
Hazardous Polymerization
Will not occur.

----------SPILL OR LEAK PROCEDURES----------

Steps to be taken if material is released or spilled:
Wear respirator, chemical safety goggles, rubber boots and heavy rubber gloves.
Sweep up, place in a bag and hold for waste disposal.
Floor may be slippery.
Avoid raising dust.
Ventilate area and wash spill site after material pickup is complete.
Waste Disposal Method:
This material may be landfilled as ordinary trash.
Observe all Federal, State, and Local Laws.

---------PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE---------

OSHA/MSHA - approved respirator.
Mechanical exhaust.
Compatible Chemical resistant gloves.
Dry ion exchange resins expand when wetted, which may cause column to shatter.

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. UMPQUA RESEARCH COMPANY SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT.

URC 80145

APPENDIX B

Document #: 80134
Revision: ____
Date Released: _____

UNIBED SORBENT SIZING CALCULATIONS

OF THE UREASE UNIBED

FOR THE

NASA-MSFC PHASE II SBIR - DEVELOPMENT OF

BIOCATALYTIC REACTORS FOR REMOVAL OF VOLATILE

CONTAMINANTS FROM PMMS, ECLSS AND LIFE SCIENCE WASTEWATER

Prepared By: _____

Approved By: _____ **Date:** _____

Approved By: _____ **Date:** _____

URC 80134

TABLE OF CONTENTS

LIST OF FIGURES AND TABLES

1.0 INTRODUCTION

This document presents the design for the Urease Unibed -- a prototype Biocatalytic Reactor used for the removal of urea. Urea is a low molecular weight compound that is not removed from dilute aqueous solution by adsorption. In this bed, it is biochemically transformed to adsorbable species that are subsequently removed by ion-exchange media. The adsorption media section of the unibed is configured for shorter life than the section containing catalyst material. Thus, breakthrough is detectable by an increase in conductivity. This enzyme unibed also imparts residual iodine into the effluent water, using iodinated resin.

The urease unibed should be installed downstream of a conventional unibed train in order to preclude or minimize interference from other waste water contaminants (see Figure 1). When it is paired with an alcohol oxidase unibed, it should precede that bed because urease is more robust than alcohol oxidase.

1.1 Applicable Documents

1.1.1 SBIR Phase II Contract NAS8-38421

1.2 Applicable Drawings

1.2.1 Umpqua Research: URC DWG 90136

1.3 General Approach

The design is based on (1) laboratory data collected by small column testing on immobilized enzyme columns at UMPQUA between January 1988 and January 1991 under NASA contracts NAS8-37642 and NAS8-38421 and (2) isotherm data from shaker table and small column single contaminant, single media tests performed at UMPQUA under the following NASA contracts:

NAS9-17073	NAS9-17464
NAS9-17523	NAS9-17611

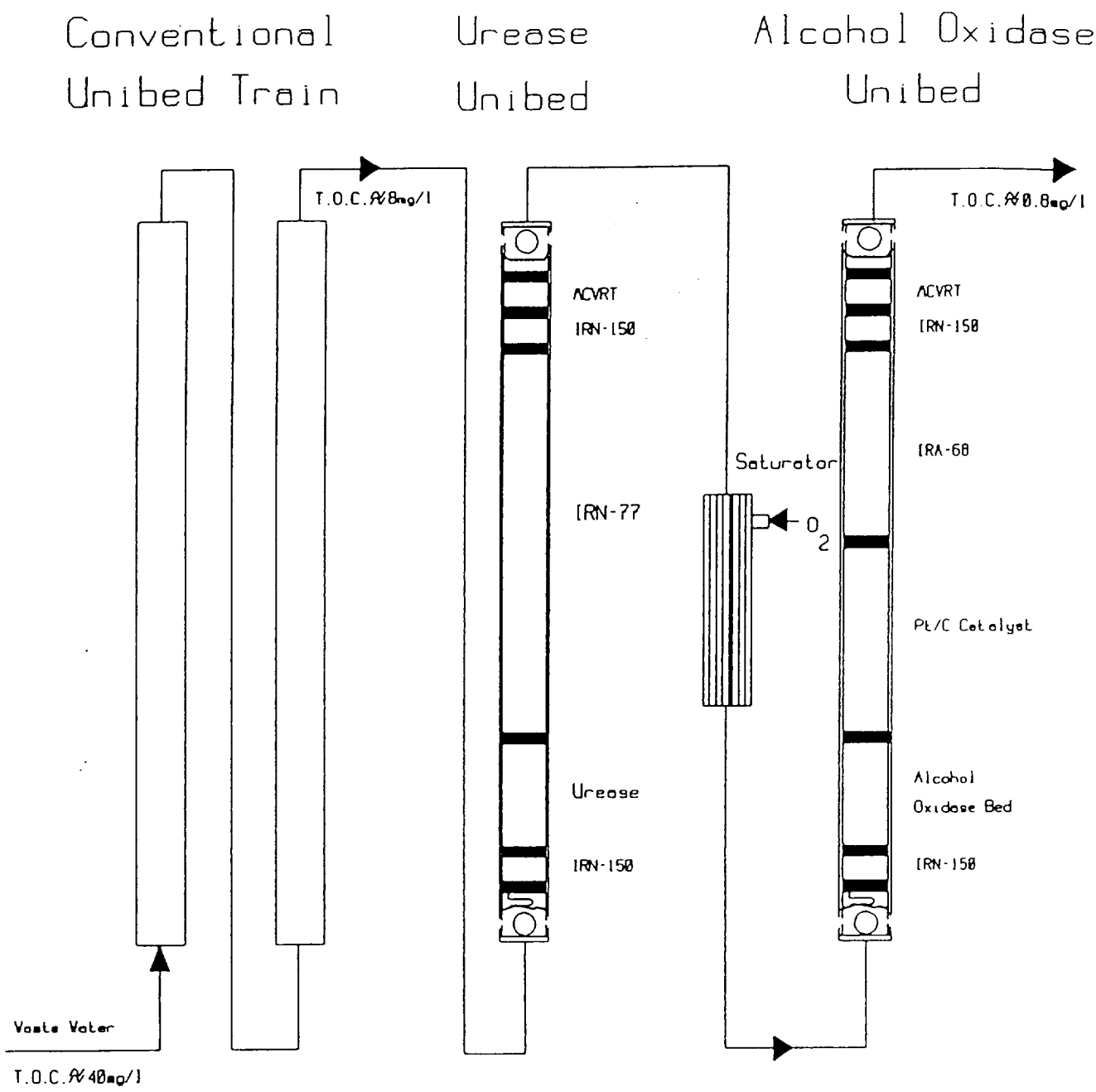


Figure 1 Location of Enzyme Unibeds

2.0 DESIGN REQUIREMENTS

2.1 Configuration

2.1.1 One 24 in long, 2 in diameter polycarbonate tube with an inlet and an outlet Quick Disconnect (QD).

2.2 Life at Design Conditions

2.2.1 Throughput: 504 L

2.2.2 Time: 14 days (limited by the quantity of ion-exchange material in this demonstration unit)

2.3 Inlet Solution

2.3.1 Waste Hygiene Water Effluent from Hygiene Unibeds (URC #90087).

2.3.2 Urea: 60 mg/L

2.4 Flow

2.4.1 Flow Rate: 25 mL/min ≈ 1.5 L/hr (3.3 lb/hr)

2.4.2 Daily Operating Time: 24 hr/day

2.4.3 1-Day Thruput: 36 L

2.5 Temperature

2.5.1 Operating Range: 68 - 77 F

2.6 Pressure

2.6.1 Maximum Operating Pressure (MOP): 40 psig

2.6.2 Proof Pressure: 60 ± 5 psig

2.7 Pressure Drop

2.7.1 Maximum Allowable Pressure Drop: 5 psig

2.8 Iodine Output

2.8.1 Range: 0.5 - 6.0 ppm

2.9 Outlet Quality

2.9.1 Water Quality Requirements: See Table 1. (NOTE: This standard applies prior to iodination.)

3.0 DESIGN DATA

The design data were developed by UMPQUA under contract to NASA-MSFC for the enzyme portion of the unibed and under contract to NASA-JSC for the ion exchange and MCV media (see Paragraph 1.3 for applicable contract numbers).

3.1 Sorbent Selection

The best performing media have been selected for each bed, based on single adsorbent-single contaminant/shaker table and single adsorbent-single contaminant/dynamic column tests run previously by UMPQUA. The selected adsorbents are listed in Table 2.

3.1 Adsorption Equilibrium Data

Table 2 also contains ion exchange loadings (equilibrium data) needed for the design of the sorption sub-beds. These data are from UMPQUA small-column tests and are lower than the manufacturer's published values.

4.0 UNIBED DESIGN

4.1 Unibed Dimensions

Each unibed consists of a single 2 in x 24 in long polycarbonate housing containing nominally, 1100 cc of media. The total bed length is 22 in. A sub-bed volume of 60 cc is the minimum bed length to diameter ratio necessary to insure proper sub-bed performance. The remaining volume is occupied by lip seals, an internal spring and the end caps.

TABLE 1. WATER QUALITY REQUIREMENTS
(Maximum Contaminant Levels)

QUALITY PARAMETERS	HYGIENE WATER
PHYSICAL PARAMETERS	
Total Solids (mg/l)	500
Color, True (Pt/Co units)	15
Taste (TTN)	N/A
Odor (TON)	3
Particulates (max size - microns)	40
pH	5.0-8.5
Turbidity (NTU)	1
Dissolved gas (free @ 37 C)	N/A
Free gas (@ STP)	Note 1
INORGANIC CONSTITUENTS (mg/l) (See Note 2)	
Ammonia	0.5
Arsenic	0.01
Barium	1.0
Cadmium	0.005
Calcium	30
Chloride	200
Chromium	0.05
Copper	1.0
Iodine (Total-includes organic iodine)	15
Iron	0.3
Lead	0.05
Magnesium	50
Manganese	0.05
Mercury	0.002
Nickel	0.05
Nitrate (NO_3-N)	10
Potassium	340
Selenium	0.01
Silver	0.05
Sulfate	250
Sulfide	0.05
Zinc	5.0

TABLE 1. WATER QUALITY REQUIREMENTS (Continued)
(Maximum Contaminant Levels)

QUALITY PARAMETERS	HYGIENE WATER
ASTHETICS (mg/l)	
Cations	N/A
Anions	N/A
CO_2	N/A
MICROBIAL	
Bacteria (CFU/100 ml)	
Total Count	1
Anaerobes	1
Coliform	1
Virus (PFU/100 ml)	1
Yeast & Mold (CFU/100 ml)	1
RADIOACTIVE CONSTITUENTS (pCi/l)	Note 3
ORGANIC PARAMETERS (µg/l) (See Note 2)	
Total Acids	500
Cyanide	200
Halogenated Hydrocarbons	10
Phenols	1
Total Alcohols	500
Total Organic Carbon (TOC)	10,000
Uncharacterized TOC (UTOC)(See Note 4)	1,000
ORGANIC CONSTITUENTS (mg/l) (See Note 2)	

Note 1: No detectable gas using a volumetric gas vs fluid measurement system. Excludes CO_2 used for aesthetic purposes.
Note 2: Each parameter/constituent MCL must be considered individually and independently of others.
Note 3: The maximum contaminant levels for radioactive constituents in potable and personal hygiene water shall conform to Nuclear Regulatory Commission (NRC) regulations (10CFR20, et al.). These maximum contaminant levels are listed in the Federal Register, Vol. 51, No. 6, 1986, Appendix B, as Table 2 (Reference Level Concentrations) Column 2 (Water). Control/contaminant/monitoring of radioactive constituents used on SSF shall be the responsibility of the user. Prior to the introduction of any radioactive constituents on SSF, approval shall be obtained from the Radiation Constraints Panel (RCP). The RCP will approve or disapprove proposed monitoring and decontamination procedures on a case-by-case basis.
Note 4: UTOC equals TOC minus the sum of analyzed organic constituents expressed in equivalent TOC.

TABLE 2. SORPTION EQUILIBRIUM DESIGN VALUES

Contaminant	Media	Mfg Capacity meq/cc	URC Design Capacity mg/cc	Maximum Swelling
Ammonium Ion	IRN-77	1.9	25	- 5%
Iodine (as I_2)	IRN-150	0.8	158	- 20%

TABLE 3. UREASE UNIBED MEDIA CONFIGURATION

Flow Direction	Sorbent	Ref. Para	Volume (cc)	Life for Urea at 60 mg/L (Days)	Function
↓	IRN-150	4.2.1	60	44	Iodine removal
	Urease	4.2.2	200	166	Urea to ammonia
	IRN-77	4.2.3	750	14	Remove NH_3 as NH_4^+
	IRN-150	4.2.4	60	44	Protect from I_2 backflow
	MCV-RT	4.2.5	60	83	Microbial control
			1130 (1 tube)	14 days	

4.2 Unibed Configuration and Sub-bed Sizing

The configuration of the urease unibed is shown in Table 3. The initial IRN-150 sub-bed functions to remove iodine imparted into solution from a conventional unibed train. The biocatalyst urease sub-bed decomposes urea into ammonia. The IRN-77 sub-bed serves to remove this ammonia from the waste water stream. Following this, a second sub-bed of IRN-150 resin protects the enzyme sub-bed from iodine in a reverse-flow situation. Finally, an MCV-RT resin sub-bed imparts iodine into the flow stream for microbial control. Since the waste water first passes through a conventional unibed assembly, the amount of ion-exchange media is sized solely for ammonia, the major product of the enzyme sub-bed. The sizing rationale for each sub-bed is presented in the following paragraphs.

4.2.1 IRN-150

Waste water enters the urease unibed from a conventional unibed (see Figure 1). The water contains residual iodine, which impairs the activity of urease. Therefore, a 60 cc bed of IRN-150 is used to remove iodine. The minimum UMPQUA working capacity for IRN-150 is 158 mg I_2/cc.

Total Sorption Capacity: 60 cc IRN-150 x 158 mg I_2/cc= 9480 mg I_2

MCV^{RT} puts out 0.5 - 6 ppm I_2

Throughput Capacity: 9480 mg I_2 + 6 mg I_2/L = 1580 L

Life: 1580 L + 36 L/day = 44 days

4.2.2 Urease.

A 6 cc bed of immobilized urease tested with 60 mg/L urea at a flow rate of 2.5 mL/min performed well up to a throughput of 40 L/cc, converting over 98% of the urea to ammonia during the first 30 L/cc. The empty-bed contact time for

this test column was 2.4 min. At the design flow of 25 mL/min, the column volume would be 60 cc to maintain a 2.4 min contact time. The urease sub-bed volume was conservatively set at 200 cc, with an empty-bed contact time of 8 minutes at 25 mL/min. At 60 mg/L urea, the throughput capacity of the enzyme sub-bed is considered to be 30 L/cc.

Life: 200 cc x 30 L/cc ÷ 36 L/day = 166 days.

Concentrations of urea higher than 60 mg/L can be hydrolyzed by this unibed.

4.2.3 IRN-77

Cations are removed by IRN-77, a strongly acidic cation exchange resin prepared by UMPQUA in the H^+ form. The ammonia product from urea is removed as the ammonium ion (NH_4^+) by this resin, which has a capacity of 25 mg NH_4^+/cc. For the hydrolysis of urea by the urease enzyme, the chemical equation is:

$$NH_2CONH_2 + H_2O \rightarrow 2NH_3 + CO_2$$

The primary product that must be removed is ammonium ion, NH_4^+:

$$NH_3 + H_2O \rightarrow NH_4^+ + OH^-$$

60 mg/L ÷ 60 mg Urea/mmol = 1.0 mmol Urea/L

$$1.0 \text{ mmol Urea/L} \times \frac{2 \text{ mmol } NH_4^+}{1 \text{ mmol Urea}} = 2.0 \text{ mmol } NH_4^+/L$$

$$2.0 \text{ mmol } NH_4^+/L \times \frac{19 \text{ mg } NH_4^+}{\text{mmol } NH_4^+} = 38 \text{ mg } NH_4^+/L$$

The 750 cc bed of IRN-77 will hold:

Total Sorption Capacity: 750 cc x 25 mg NH_4^+/cc = 18,750 mg NH_4^+

Throughput Capacity: 18,750 mg NH_4^+ ÷ 38 mg NH_4^+/L = 493 L

Life: 493 L ÷ 36 L/day = 14 days

The lifetime of the urease unibed is limited by the amount of IRN-77 used to collect the NH_4^+ product. The expected lifetime is 14 days.

4.2.4 IRN-150

This 60 cc bed precedes the MCV^{RT} resin bed and serves to catch any iodine that may be emitted during a back flow situation. The capacity of this sub-bed would be the same as calculated in 4.2.1. Its life is 44 days.

4.2.5 MCV^{RT} Resin.

MCV^{RT} resin is required at the exit of each unibed for microbial control. The resin puts out 0.5 - 6 ppm I_2 for a duration of 50 L/cc media. The lifetime of this sub-bed is:

Life: 60 cc x 50 L/cc ÷ 36 L/day = 83 days

4.2.6 Sizing Discussion

The design summarized in Table 3 was obtained within the dimension restrains given in Paragraph 4.1. The capacity is limited by the amount of resin in the IRN-77 sub-bed. Adding a second 1100 cc tube containing IRN-77 would increase the life of the unibed from 14 days to 35 days. A further improvement would result from adjusting the size of each sub-bed to match the 83 day life of the MCV-RT resin.

4.3 Pressure Drop.

Previous testing developed a pressure drop equation.

$\delta P = 0.4\ WL\ \mu/D^2$

where: δP = Pressure drop, psi

W = flow rate, lb/min

L = bed length, in

D = bed diameter, in

μ = viscosity, centipoise

For the enzyme unibeds:

W = 2.5 lb/hr = 0.0417 lb/min

L = 21.5 in

D = 2 in

μ = 1 centipoise

$\delta P = 0.4\ (0.00417)(21.5)(1)/(2)^2 = 0.9$ psi

Specified max δP = 5.0 psi

4.4 Summary of Unibed Design Values.

A summary of the design values for the enzyme beds is given in Table 4.

TABLE 4. SUMMARY OF UREASE UNIBED DESIGN VALUES

Parameter	Value
URC Drawing Number	90136
Nominal ID	2 in
Water System	Hygiene
Flow Rate	3.3 lb/hr (1.5 L/hr)
Daily Operating Time	24 hr/day
Thruput, 1 day	36 L
Total Media Volume	1130 cc
Cross Sectional Area	20.3 cm^2
Total Length of Media (Installed)	22 in
Face Velocity	1.23 cm/min
Empty Bed Contact Time - Urease Sub-bed	8 min
- Unibed	44 min
Life - (limited by IRN-77) -	504 L
-	14 days

APPENDIX I

MEDIA INFORMATION

IRN-150

IRN-77

ROHM AND HAAS COMPANY
PHILADELPHIA, PENNSYLVANIA 19105
FLUID PROCESS CHEMICALS

AMBERLITE ION EXCHANGE RESINS

AMBERLITE® IRN-150

Amberlite IRN-150 is a mixture of gelular, polystyrene cation and anion exchange resins. Amberlite IRN-150 resin as supplied contains a stoichiometric equivalent of the strongly acidic cation (Amberlite IRN-77) and the strongly basic anion (Amberlite IRN-78) exchange resins. It is supplied in the hydrogen/hydroxide form as clear, amber colored spherical particles virtually perfect in bead appearance. Amberlite IRN-150 resin is designed for use in industrial water treatment applications, particularly in once through applications such as primary water chemistry control in nuclear power operations. This resin combines the properties of high capacity and excellent resistance to bead fracture from attrition or osmotic shock.

Amberlite IRN-150 resin is designated as a Nuclear Grade resin and is manufactured using special processing procedures. These procedures, combined with a patented Rohm and Haas process to reduce the chloride content of the anion component, produce material of the ultimate purity and yield a product meeting the exacting demands of the nuclear industry. Amberlite IRN-150 resin is recommended in any non-regenerable mixed bed application where reliable production of the highest quality water is required and where the "as supplied" resin must have an absolute minimum of ionic and non-ionic contamination.

IMPORTANT FEATURES OF AMBERLITE IRN-150 ION EXCHANGE RESIN

HIGH CAPACITY: Amberlite IRN-150 resin will exhibit a nominal operating capacity of 12 kg/ft³ (0.55 meq/ml).

EXCEPTIONAL PURITY: Amberlite IRN-150 resin is manufactured to demanding purity specifications which assure a minimum of ionic and non-ionic contamination.

GOOD RESISTANCE TO BEAD FRACTURE: Amberlite IRN-150 resin offers superior performance with respect to particle breakdown from attrition or osmotic shock.

INSOLUBLE IN ALL COMMON SOLVENTS

RECOMMENDED CONDITIONS OF OPERATION

The recommended conditions for operation of Amberlite IRN-150 resin are listed below.

BED DEPTH:	24" minimum (0.61 m)
SERVICE FLOW RATE:	2–5 gpm/ft³ (16 to 40.1 l/hr/l)

PHYSICAL CHARACTERISTICS

SHAPE:	Spherical beads
SHIPPING WEIGHT:	43 lbs/ft³ (688 g/l)
PARTICLE SIZE (U.S. MESH):	

Screen Size	% Maximum
+16	5.0
−40	5.0
−50	0.5

PERFECT BEADS: 95% minimum

CHEMICAL CHARACTERISTICS

IONIC FORM:	Hydrogen/Hydroxide
CATION TO ANION EQUIVALENT RATIO:	1:1

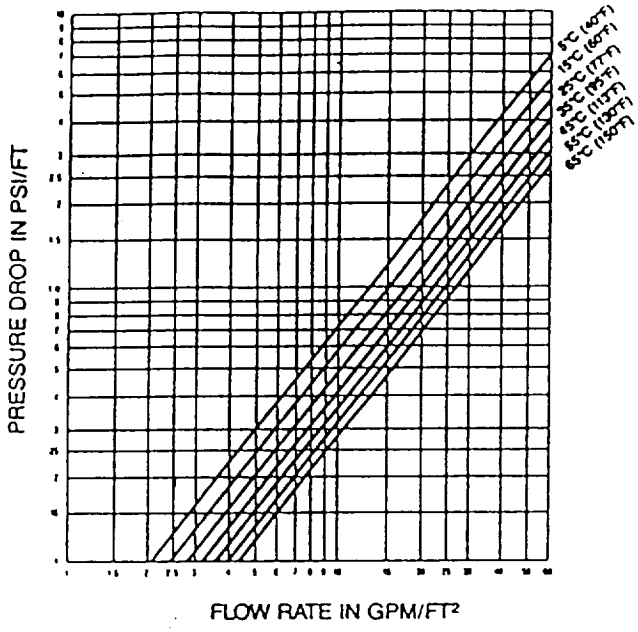
Ionic Content by Individual Component:	IRN-77	IRN-78
Equivalent % H, minimum	99.0	na
Equivalent % OH, minimum	na	95.0
Equivalent % Cl, maximum	na	0.10
Equivalent % CO_3, maximum	na	5.0
Equivalent % SO_4, maximum	na	0.10
Sodium (ppm dry resin) maximum	50	50
Iron (ppm dry resin) maximum	50	50
Copper (ppm dry resin) maximum	10	10
Heavy metals as Pb (ppm dry resin) maximum	10	10
Aluminum (ppm dry resin) maximum	50	50
Calcium (ppm dry resin) maximum	50	50
Magnesium (ppm dry resin) maximum	50	50

HYDRAULIC CHARACTERISTICS

PRESSURE DROP: The approximate pressure drop for each foot of d depth of Amberlite IRN-150 resin in normal down flow eration at various temperatures and flow rates is shown in the graph below.

RESIN HANDLING: To retain the high purity standards of iclear grade resins, deionized water should be used for all sin handling. Contact of the resin with air should also be minimized to avoid CO_2 pickup and subsequent loss of capacity of the anion resin.

AMBERLITE® IRN-150 RESIN
PRESSURE DROP



METRIC CONVERSION GPM/ft² to M hr = GPM/ft² × 2.45
PSI/ft to MH₂O/M resin = PSI/ft × 2.30

APPLICATIONS

MIXED BED DEIONIZATION: The physical and chemical characteristics of Amberlite IRN-150 resin provide excellent performance when used in production of high quality water in any mixed bed deionization application.

NUCLEAR APPLICATIONS: The purity and physical stability of Amberlite IRN-150 resin provides unsurpassed performance in nuclear applications such as chemistry control in primary water treatment. Amberlite IRN-150 resin can also be used for a variety of rad waste applications.

PRODUCTION OF ULTRA PURE WATER: Amberlite IRN-150 resin is an excellent choice for once through (non-regenerable) applications typically found in the final DI water processing for the semiconductor industry. Amberlite IRN-150 resin provides rapid rinse to 18 megohm, high capacity, and reliable production of the highest quality water.

SAFE HANDLING INFORMATION

A Material Safety Data Sheet is available for Amberlite IRN-150 resin. To obtain a copy, contact your Rohm and Haas representative.

CAUTION: Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact.

Nitric acid and other strong oxidizing agents can cause explosive type reactions when mixed with ion exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidizing agent such as nitric acid is contemplated. Before using strong oxidizing agents in contact with ion exchange resins, consult sources knowledgeable in the handling of these materials.

ROHM AND HAAS COMPANY
PHILADELPHIA, PENNSYLVANIA 19105
FLUID PROCESS CHEMICALS

AMBERLITE® IRN-77

Amberlite IRN-77 is a strongly acidic gelular polystyrene cation exchange resin supplied in the hydrogen form. This resin is Nuclear Grade and processed to the highest purity standards to meet the stringent requirements of the Nuclear Industry. Amberlite IRN-77 contains a minimum of 99% of its exchange sites in the hydrogen form.

The manufacturing process for this resin is controlled to keep inorganic impurities at the lowest possible levels. Special treatment procedures are also used to remove traces of soluble organic compounds. These high standards of resin purity will help keep nuclear systems free of contaminants and deposits, and prevent increases in radioactivity levels due to activation of impurities in the reactor core.

IMPORTANT FEATURES OF AMBERLITE IRN-77 ION EXCHANGE RESIN

HIGH CAPACITY: Amberlite IRN-77 resin exhibits a minimum capacity of 1.8 meq/ml.

EXCEPTIONAL PURITY: Amberlite IRN-77 resin is manufactured to demanding purity specifications which assure a minimum of ionic and nonionic contamination.

GOOD RESISTANCE TO BEAD FRACTURE: Amberlite IRN-77 resin offers excellent performance with respect to particle break down from attrition or osmotic shock.

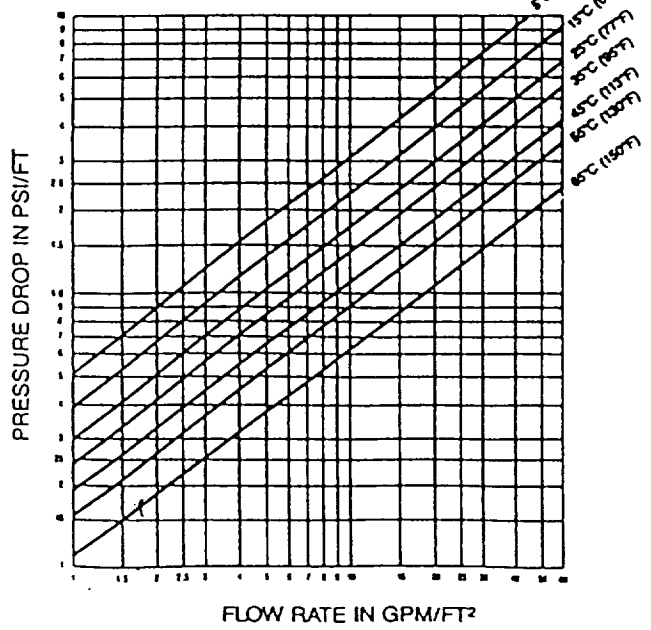
INSOLUBLE IN ALL COMMON SOLVENTS

HYDRAULIC CHARACTERISTICS

PRESSURE DROP: The approximate pressure drop for each foot of bed depth of Amberlite IRN-77 resin in normal downflow operation at various temperatures and flow rates is shown in the graphs below (data based on backwashed and classified resin bed).

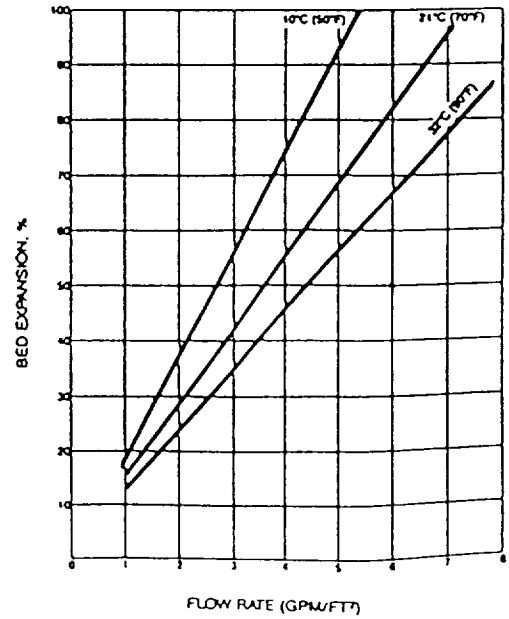
RESIN HANDLING: To retain the high purity standards of nuclear grade resins, deionized water should be used for all resin handling. If the resin requires backwashing the bed should be expanded a minimum of 50%.

AMBERLITE® IRN-77 RESIN
PRESSURE DROP



METRIC CONVERSION GPM/ft² to M hr = GPM/ft² × 2.45
PSI/ft to MH₂O/M resin = PSI/ft × 2.30

AMBERLITE® IRN-77 RESIN
HYDRAULIC EXPANSION



METRIC CONVERSION GPM/ft² to M hr = GPM/ft² × 2.45

RECOMMENDED CONDITIONS OF OPERATION

DEPTH:	24" minimum (0.61m)
RATING TEMPERATURE:	250°F maximum (121°C)
ERVICE FLOW RATE:	1–5 gpm/ft³ (8.0 to 40.1 l/hr/l)

CHEMICAL CHARACTERISTICS

ONIC FORM:	Hydrogen
AL EXCHANGE CAPACITY:	1.8 meq/ml minimum
ISTURE CONTENT:	55% maximum
ONIC CONTENT:	
uivalent % H, minimum	99
ALS CONTENT:	
Sodium, (ppm dry resin) maximum	50
on, (ppm dry resin) maximum	50
opper, (ppm dry resin) maximum	10
Heavy Metals as Pb, (ppm dry resin) maximum	10
Aluminum, (ppm dry resin) maximum	50
alcium, (ppm dry resin) maximum	50
agnesium, (ppm dry resin) maximum	50

PHYSICAL CHARACTERISTICS

HAPE:	Spherical beads
PPING WEIGHT:	50 lbs/ft³ (800g/l)
TICLE SIZE (U.S. MESH):	
creen Size	% Maximum
+16	5.0
−40	5.0
−50	0.5
HATILLON:	
., gm/bead minimum	350
00 gm/bead minimum	95
ERFECT BEADS:	95% minimum
UBILITY:	0.10% maximum

APPLICATIONS

PRIMARY WATER TREATMENT: Amberlite IRN-77 resin is very effective in removing fission products, activated corrosion products, suspended matter and Lithium 7 from reactor coolant streams.

RAD WASTE TREATMENT: Amberlite IRN-77 resin is very effective in removing radioactive cations such as Cesium 137 from waste streams.

DECONTAMINATION: Amberlite IRN-77 resin removes cationic radioactive material from spent decontaminating solutions.

SAFE HANDLING INFORMATION

A Material Safety Data Sheet is available for Amberlite IRN-77 resin. To obtain a copy, contact your Rohm and Haas representative.

CAUTION: Acidic and basic regenerant solutions are corrosive and should be handled in a manner that will prevent eye and skin contact.

Nitric acid and other strong oxidizing agents can cause explosive type reactions when mixed with ion exchange resins. Proper design of process equipment to prevent rapid buildup of pressure is necessary if use of an oxidizing agent such as nitric acid is contemplated. Before using strong oxidizing agents in contact with ion exchange resins, consult sources knowledgeable in the handling of these materials.

APPENDIX II

MATERIAL SAFETY DATA SHEETS

IRN-150

UREASE BED

IRN-77

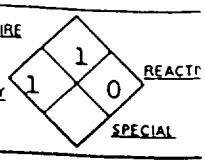
MCV-RT IODINATED RESIN

ROHM AND HAAS COMPANY

CORPORATE PRODUCT INTEGRITY DEPARTMENT
INDEPENDENCE MALL WEST
PHILADELPHIA, PA 19105

EMERGENCY TELEPHONE
215-592-3000 (ROHM AND HAAS)
800-424-9300 (CHEMTREC)

HAZARD RATING
4=EXTREME
3=HIGH
2=MODERATE
1=SLIGHT
0=INSIGNIFICANT
**=SEE SECTION IV



BS242
LIST 7

MATERIAL SAFETY DATA SHEET

NOT OSHA HAZARDOUS
NOT WHMIS CONTROLLED

MATERIAL	CODE	KEY	DOT HAZARD CLASS
AMBERLITE® IRN-150 Resin	69855	891090-3	NON-REGULATED
	DATE ISSUED 11/08/88		

FORMULA	CHEMICAL NAME OR SYNONYMS
Not applicable	Mixed bed ion exchange resin (hydrogen and hydroxide forms)

I – COMPOSITIONAL INFORMATION

	CAS Reg. No.	APPROX WT %	TWA/TLV R&H	OSHA	ACGIH
Anion/cation exchange resin	NONHAZ	35-50	NE	NE	NE
Water	NONHAZ	50-65	NE	NE	NE

NE = None established

II – PHYSICAL PROPERTY INFORMATION

APPEARANCE - ODOR - pH.			VISCOSITY
Beads; pH (aqueous slurry) = 5 to 9			NA
MELTING OR FREEZING POINT	**BOILING POINT**	**VAPOR PRESSURE (mm Hg)**	**VAPOR DENSITY (AIR=1)**
0C/32F (water)	100C/212F (water)	17 @20C (water)	Less than 1 (water)
SOLUBILITY IN WATER	**PERCENT VOLATILE (BY WEIGHT)**	**SPECIFIC GRAVITY (WATER=1)**	**EVAPORATION RATE (BUTYL ACETATE=1)**
Negligible	50-65 (water)	1.1-1.3	Less than 1 (water)

III – FIRE AND EXPLOSION HAZARD INFORMATION

FLASH POINT	AUTO IGNITION TEMPERATURE	LOWER EXPLOSION LIMIT (%)	UPPER EXPLOSION LIMIT (%)
NA	500C/932F (est.)	NA	NA

EXTINGUISHING MEDIA

☐ FOAM ☐ "ALCOHOL" FOAM ☒ CO_2 ☒ DRY CHEMICAL ☒ WATER SPRAY ☐ OTHER

SPECIAL FIRE FIGHTING PROCEDURES

Wear self-contained breathing apparatus (pressure-demand, MSHA/NIOSH-approved or equivalent) and full protective gear.

UNUSUAL FIRE AND EXPLOSION HAZARDS

Toxic combustion products may include alkylamines and oxides of sulfur and nitrogen.

IV – HEALTH HAZARD INFORMATION

ROHM AND HAAS RECOMMENDED WORK PLACE EXPOSURE LIMITS

STEL = None established.

EFFECTS OF OVEREXPOSURE

Eye Contact: Product can cause eye irritation.

EMERGENCY AND FIRST AID PROCEDURES

Eye Contact: Immediately flush eyes with large amounts of water and continue for at least 15 minutes. Get prompt medical attention.

FORM 9706 REV. 11/83

V – REACTIVITY INFORMATION

STABILITY	CONDITIONS TO AVOID
[X] STABLE [ ] UNSTABLE	Temperatures over 200C/392F.

HAZARDOUS DECOMPOSITION PRODUCTS Thermal decomposition may yield styrene monomer, divinylbenzene, alkylamines and oxides of sulfur and nitrogen.

HAZARDOUS POLYMERIZATION	CONDITIONS TO AVOID
[ ] MAY OCCUR [X] WILL NOT OCCUR	None known

INCOMPATIBILITY (MATERIALS TO AVOID) [ ] WATER [X] OTHER Avoid contact with concentrated nitric acid or any other strong oxidizing agent at all times.

VI – SPILL OR LEAK PROCEDURE INFORMATION

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED

Floor may be slippery. Use care to avoid falls. Sweep up and transfer to containers for recovery or disposal.

WASTE DISPOSAL METHODS Unused resin may be incinerated or landfilled in facilities meeting local, state and federal regulations. For contaminated resin, the user must determine the hazard and use an appropriate disposal method.

VII – SPECIAL PROTECTION INFORMATION

VENTILATION TYPE

Normal room ventilation.

RESPIRATORY PROTECTION

None required for normal operations.

PROTECTIVE GLOVES	EYE PROTECTION
None required	Safety glasses (ANSI Z-87.1 or approved equivalent)

OTHER PROTECTIVE EQUIPMENT

Eyewash facility

VIII – STORAGE AND HANDLING INFORMATION

STORAGE TEMPERATURE	INDOOR	HEATED	REFRIGERATED	OUTDOOR
MAX. 49C/120F MIN. 0C/32F	YES	NO	NO	YES

NOTE: Store at ambient temperatures. Avoid repeated freeze-thaw cycles.

NOTE: Ground ion exchange resins should be treated as potential eye irritants. A finely ground form of a structurally related strong acid cation exchange resin produced severe rabbit eye irritation.

NOTE: The maximum operating temperature for this product is 60C/140F. Functional group destruction and loss of capacity will occur above this temperature.

IX – TOXICITY INFORMATION

No toxicity data available for this product.

X – MISCELLANEOUS INFORMATION

Caution: Do not pack column with dry ion exchange resins. Dry beads expand when wetted; this expansion can cause a glass column to shatter.

Caution: Nitric acid and other strong oxidizing agents can cause explosive-type reactions when mixed with ion exchange resins. Proper design of equipment to prevent rapid build-up of pressure is necessary if use of an oxidizing agent such as nitric acid is contemplated. Before using strong oxidizing agents in contact with ion exchange beads, consult sources knowledgeable in handling these materials.

NA = NOT APPLICABLE C = CEILING VALUE	KEY	DATE OF ISSUE	SUPERSEDES
	891090-3	11/08/88	09/04/87

MATERIAL SAFETY DATA SHEET

Umpqua Research Company
P.O. Box 791 - 125 Volunteer Way
Myrtle Creek, OR 97457
(503) 863-7770

Feb. 25, 1991

----------IDENTIFICATION----------

PRODUCT #: 90021-55 **NAME:** Urease Bed

----------HAZARDOUS INGREDIENTS----------

Urease enzyme on a silica base.

----------PHYSICAL/CHEMICAL CHARACTERISTICS----------

Orange powder or granules; free flowing when dry.

----------FIRE AND EXPLOSION HAZARD DATA----------

Avoid open flames. May emit toxic fumes under fire conditions. Wear self-contained breathing apparatus. The silica base is non-flammable. Enzyme fire may be extinguished using water, foam, dry chemical or CO_2 extinguisher.

----------REACTIVITY DATA----------

Avoid close proximity to volatile solvents. Do not contact with reducing agents or strong oxidizing agents.

----------HEALTH HAZARD DATA----------

This product contains crystalline silica, which is considered a hazard by inhalaton. IARC has classified crystalline silica as a probable carcinogen for humans, although NTP and OSHA have not. Silica is also a know cause of silicosis, a non-cancerous lung disease, when inhaled repeatedly at high levels.

The urease enzyme may be harmful if ingested, and the polypeptide material may cause allergic sensitization upon repeated skin contact.

----------PRECAUTIONS FOR SAFE HANDLING AND USE----------

Store away from volatile organic solvents. Store wet, under water. Do not ingest or inhale. Avoid eye contact.

----------CONTROL MEASURES----------

Use rubber gloves and wear goggles when handling. If spilled, clean up with broom and dustpan. This material may be land-filled as ordinary trash. Avoid raising dust.

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. UMPQUA RESEARCH COMPANY SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT.

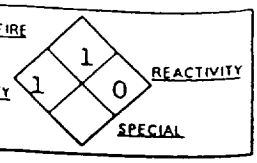
URC 80148

ROHM AND HAAS COMPANY

CORPORATE PRODUCT INTEGRITY DEPARTMENT
INDEPENDENCE MALL WEST
PHILADELPHIA, PA 19105

EMERGENCY TELEPHONE
215-592-3000 (ROHM AND HAAS)
800-424-9300 (CHEMTREC)

HAZARD RATING
4=EXTREME
3=HIGH
2=MODERATE
1=SLIGHT
0=INSIGNIFICANT
*=SEE SECTION IV



BS231
LIST 7

MATERIAL SAFETY DATA SHEET

NOT OSHA HAZARDOUS
NOT WHMIS CONTROLLED

MATERIAL	CODE	KEY	DOT HAZARD CLASS
AMBERLITE® IRN-77 Resin	69213	906197-1	NONREGULATED
	DATE ISSUED 11/02/88		

FORMULA	CHEMICAL NAME OR SYNONYMS
Not applicable	Strong acid cation exchange resin (hydrogen form)

I – COMPOSITIONAL INFORMATION

	CAS REG. NO.	APPROX WT %	TWA/TLV R&H	OSHA	ACGIH
Styrene/divinylbenzene cation exchange resin	NONHAZ	40-50	NE	NE	NE
Water	NONHAZ	50-60	NE	NE	NE

NE = None established

II – PHYSICAL PROPERTY INFORMATION

APPEARANCE - ODOR - pH.			VISCOSITY
Beads; pH (aqueous slurry) 3.0 max.			NA
MELTING OR FREEZING POINT	**BOILING POINT**	**VAPOR PRESSURE (mm Hg)**	**VAPOR DENSITY (AIR=1)**
0C/32F (water)	100C/212F (water)	17 @ 20C/68F	Less than 1
SOLUBILITY IN WATER	**PERCENT VOLATILE (BY WEIGHT)**	**SPECIFIC GRAVITY (WATER=1)**	**EVAPORATION RATE (BUTYL ACETATE=1)**
Negligible	50-60 (water)	1.1-1.4	Less than 1

III – FIRE AND EXPLOSION HAZARD INFORMATION

FLASH POINT	AUTO IGNITION TEMPERATURE	LOWER EXPLOSION LIMIT (%)	UPPER EXPLOSION LIMIT (%)
NA	500C/932F (est.)	NA	NA

EXTINGUISHING MEDIA

☐ FOAM ☐ "ALCOHOL" FOAM ☒ CO_2 ☒ DRY CHEMICAL ☒ WATER SPRAY ☐ OTHER

SPECIAL FIRE FIGHTING PROCEDURES

Wear self-contained breathing apparatus (pressure-demand, MSHA/NIOSH-approved or equivalent) and full protective gear.

UNUSUAL FIRE AND EXPLOSION HAZARDS

Toxic combustion products include oxides of sulfur.

IV – HEALTH HAZARD INFORMATION

ROHM AND HAAS RECOMMENDED WORK PLACE EXPOSURE LIMITS

STEL = None established

EFFECTS OF OVEREXPOSURE

Eye Contact: Product, as supplied, can cause eye irritation.

EMERGENCY AND FIRST AID PROCEDURES

Eye Contact: Flush eyes with large amounts of water for at least 15 minutes. Get prompt medical attention.

FORM SPM REV 11/87

V – REACTIVITY INFORMATION

STABILITY	CONDITIONS TO AVOID
[X] STABLE [ ] UNSTABLE	Temperatures over 200C/392F.

HAZARDOUS DECOMPOSITION PRODUCTS
Thermal decomposition may yield styrene monomer, divinylbenzene, and sulfur oxides.

HAZARDOUS POLYMERIZATION	CONDITIONS TO AVOID
[ ] MAY OCCUR [X] WILL NOT OCCUR	None known

INCOMPATIBILITY (MATERIALS TO AVOID) [ ] WATER [X] OTHER
Avoid contact with concentrated nitric acid or any other strong oxidizing agents at all times.

VI – SPILL OR LEAK PROCEDURE INFORMATION

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED
Floor may be slippery. Use care to avoid falls. Sweep up and transfer to containers for recovery or disposal.

WASTE DISPOSAL METHODS Unused resin may be incinerated or landfilled in facilities meeting local, state and federal regulations. For contaminated resin, the user must determine the hazard and use an appropriate disposal method.

VII – SPECIAL PROTECTION INFORMATION

VENTILATION TYPE
Normal room ventilation.

RESPIRATORY PROTECTION
None required for normal operations.

PROTECTIVE GLOVES	EYE PROTECTION
None required	Safety glasses (ANSI Z-87.1 or approved equivalent)

OTHER PROTECTIVE EQUIPMENT
Eyewash facility

VIII – STORAGE AND HANDLING INFORMATION

STORAGE TEMPERATURE	INDOOR	HEATED	REFRIGERATED	OUTDOOR
MAX. 60C/140F MIN. 0C/32F	YES	NO	NO	YES

NOTE: Store at ambient temperatures. Avoid repeated freeze-thaw cycles.
NOTE: Ground ion exchange resins should be treated as potential eye irritants. A finely ground form of a structurally related strong acid cation exchange resin produced severe rabbit eye irritation.
NOTE: The maximum operating temperature for this product is 121C/250F. Functional group destruction and loss of capacity will occur above this temperature.

IX – TOXICITY INFORMATION

No toxicity data available for this product.

X – MISCELLANEOUS INFORMATION

Caution: Do not pack column with dry ion exchange resins. Dry beads expand when wetted; this expansion can cause a glass column to shatter.
Caution: Nitric acid and other strong oxidizing agents can cause explosive-type reactions when mixed with ion exchange resins. Proper design of equipment to prevent rapid build-up of pressure is necessary if use of an oxidizing agent such as nitric acid is contemplated. Before using strong oxidizing agents in contact with ion exchange beads, consult sources knowledgeable in handling these materials.

NA = NOT APPLICABLE C = CEILING VALUE	KEY	DATE OF ISSUE	SUPERSEDES
	906197-1	11/02/88	07/31/87

UMPQUA RESEARCH COMPANY
P.O. BOX 791 - 626 N.E. DIVISION
MYRTLE CREEK, OREGON 97457
(503) 863-5201
FAX (503) 863-6199

MATERIAL SAFETY DATA SHEET

----------IDENTIFICATION----------

PRODUCT #: 90021-47 NAME: MCV-RT Iodinated Resin

----------TOXICITY HAZARDS----------

Effects of Overexposure: Can irritate eyes, nose, throat and skin, hypersensitivity, nausea, abdominal pain, diarrhea, excessive thirst, circulatory failure. Possibly fatal if swallowed.

----------HEALTH HAZARD DATA----------

Threshold Limit Value
TVL-air: 0.1 ppm as Iodine TXDS: orl-Hmn LDLo: 5 mg/kg as Iodine

First Aid Procedures:

Skin: wash with soap/water; get medical assistance.

Eyes: flush thoroughly with water 15 minutes. Assure adequate flushing by separating the eyelids with fingers; get medical assistance.

Inhalation: remove to fresh air; get medical assistance.

Ingestion: give milk, starch solution, or tablespoon sodium thiosulfate in a glass of water and get immediate medical attention. Treat for shock.

Acute Effects: may cause eye irritation. Particles can irritate the eyes. Finely ground particles of similar material caused corneal damage in rabbit eyes.

----------PHYSICAL DATA----------

Specific Gravity: 1.11

Appearance and Odor: Dark purple to black beads, with moderate iodine and amine odor.

Solubility: Beads release iodine in water in concentrations below 300 ppm

----------FIRE AND EXPLOSION HAZARD DATA----------

Autoignition Temperature: 427 C EST

Extinguishing Media
Carbon Dioxide
Dry Chemical Powder
Water Spray

Special Firefighting Procedures
Wear Self-contained breathing apparatus and protective clothing to prevent contact with skin and eyes.

Unusual Fire and Explosions hazards
Emits Toxic fumes under fire conditions.

URC 80145

UMPQUA RESEARCH COMPANY
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MYRTLE CREEK, OREGON 97457
(503) 863-5201
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MATERIAL SAFETY DATA SHEET

--------IDENTIFICATION--------

PRODUCT #: 90021-47 NAME: MCV-RT Iodinated Resin

----------REACTIVITY DATA----------

Drying results in release of iodine vapor.
Stability: stable.
Conditions to avoid: Temperatures over 220 C.

Incompatibilities: Nitric Acid and other strong Oxidizing agents can cause explosion.
Materials to avoid: NH_3, Acetylene, Acetaldehyde, Active metals particularly powdered Al.
Reactions when mixed with ion exchange resins.
Hazardous combustion or decomposition products.
Styrene Monomer, Divinylbenzene
Toxic fumes of:
Carbon Monoxide and Carbon Dioxide
Nitrogen Oxides
Hazardous Polymerization
Will not occur.

----------SPILL OR LEAK PROCEDURES----------

Steps to be taken if material is released or spilled:
Wear respirator, chemical safety goggles, rubber boots and heavy rubber gloves.
Sweep up, place in a bag and hold for waste disposal.
Floor may be slippery.
Avoid raising dust.
Ventilate area and wash spill site after material pickup is complete.
Waste Disposal Method:
This material may be landfilled as ordinary trash.
Observe all Federal, State, and Local Laws.

---------PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE---------

OSHA/MSHA - approved respirator.
Mechanical exhaust.
Compatible Chemical resistant gloves.
Dry ion exchange resins expand when wetted, which may cause column to shatter.

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. UMPQUA RESEARCH COMPANY SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT.

JRC 80145

APPENDIX C

Appendix C: Conditions for Alcohol Oxidase Test Columns.

The alcohol oxidase enzyme was isolated from pichia yeast, and used as a sucrose solution containing 1000 EU/mL. One EU is defined as the amount of enzyme which will produce one millimole of acetaldehyde at pH=7, 25°C. Generally, each derivatized support required one milliliter enzyme per gram dry weight of support.

All column testing used 20 PPMV ethanol as the challenge solution unless otherwise noted. Small column tests (<1 cc/bed) were conducted with gravity feeds for several different modifications of the preparation method. When these tiny column tests showed exceptional abilities to convert ethanol to acetaldehyde, larger scale small column tests were conducted. The test conditions and results of all of these small column tests are discussed in the paragraphs below.

I. Alcohol Oxidase column 900104 was started on 1/4/90 and run at a flow rate of 1.9 mL/min. This 9.3 cc column ran for 0.95 L/cc and was not very effective at converting ethanol to acetaldehyde.

II. Column 11AOR was started on 3/29/90 and operated at a flow rate of 5.0 mL/min. This 9.2 cc bed was prepared by the standard titanium method with full dilutions of all reagents as described in the Phase I procedure. This method uses 1% hexane-

diamine as the linkage agent. Enzyme incubation occurs at room temperature. A tris buffer wash was used after immobilization to rinse off unbound enzyme. The column ran for 0.3 L/cc and removed less than half the alcohol initially.

Column 11AO was run concurrently and operated at the same flow rate. The 9.6 cc bed was also prepared by the dilute titanium method, but was not rinsed with tris buffer. This column also was run for only 0.3 L/cc, since it was ineffective.

III. Columns 16N and 16O were started on 4/10/90. Each 5.0 cc bed was run at a flow rate of 2.0 mL/min. Both were prepared by the dilute titanium method, the difference being that the alcohol oxidase enzyme was from different batches. The 16N column used a freshly-thawed deep red batch of enzyme whereas the 16O column was prepared from yellow enzyme which had been stored at 4° C for an indefinite amount of time. The test columns ran for 1.2 L/cc. Both columns oxidized all the ethanol at the start of the test, but were ineffective by the end of the test. The 16N column was more efficient than the 16O bed.

IV. Column 24Pt, started on 4/17/90, contained a 7.6 cc bed which was run at a 2.0 mL/min flow rate. The support had platinum reduced on at a concentration equal to 0.55% of the total support weight. The preparation was by the silanization method, using 25% g-aminopropyltriethoxysilane as the binding agent. This method is also described in the Phase I report. At the final stage of the preparation, five mL alcohol oxidase (5000 EU) was added to five grams of derivatized support and allowed to incubate at room

temperature for 65 hours. The column had a total throughput of 8 L/cc, which was quite a bit more effective than the previous alcohol oxidase column tests.

V. Column 26HAO, which was started on 4/20/90, contained a 5.0 cc enzyme bed. The column was operated at a flow rate of 2.0 mL/min for 0.9 L/cc. The column served as a batch test of the support that was used for immobilization of several other enzymes. The column did not function effectively, and oxidized very little ethanol after setting over a weekend.

VI. Four columns were assembled on 4/20/90 to test the ability of coenzymes to remove the hydrogen peroxide byproduct of the alcohol oxidase enzyme. Alcohol oxidase bed material was prepared by the dilute titanium method. Superoxide dismutase (10 mg/30,000 EU), catalase (10 mg/28,000 EU) and horseradish peroxidase (50 mg/10,500 EU) were added to 1 g support derivatized by the dilute titanium method. The coenzyme and oxidase supports were mixed together at a level of 10% coenzyme.

Minicolumns were run to demonstrate the effectiveness of the approach. Small column tests were conducted with 8.5 cc bed volumes, at 2.0 mL/min. A control column with only alcohol oxidase enzyme was also run. The 30SOD and 30CAT columns ran for 2.6 L/cc each and were effective for only a short duration. The 30HP ran for 3.2 L/cc and was slighly better. All three were out-performed by the control column which ran for 4.2 L/cc.

VII. Column 33Pt contained 1.2% Pt reduced onto the support prior to immobilization by the dilute titanium process. The enzyme

incubation took place for 96 hours at room temperature. The 6.7 cc bed was operated at a flow rate of 2.0 mL/min. The column was started on 5/7/90 and ran for 35 L/cc. This column showed a significant improvement from any column that had been previously tested. Although the duration of complete removal of ethanol was short (about 2 L/cc), the column functioned with gradual breakdown for a 5 month period. This column is discussed in the main text.

VIII. A pair of columns (46B and 47D) were run to provide a direct comparison of the silane and dilute titanium processes. Two samples of Pt deposited support were taken through each process of immobilization. The silane column (46B) also was compared to a silane column containing no Pt (49S). All three columns were started on 5/24/90, and were operated at 2.0 mL/min. The column volumes were 6.4 cc for 46B, 5.1 cc for 47D and 5.7 cc for 49S.

The results showed that 47D was the most effective column for diminishing the ethanol content of the challenge solution. Surprisingly, the silane control column worked better than the platinum column, although neither column was terribly efficient. It should be noted that the incubation time for the 49S column was 21 days, as opposed to 120 hours for the other two columns. The result strongly implies that the longer the incubation period, the better the immobilization process. A minimum 96 hour incubation period was used hereafter.

IX. Two columns (61L and 61M) were set up to address the problem of complete organic removal from the challenge stream. Rather than monitor the convertion of ethanol to acetaldehyde,

these columns were intended to remove the product from the solution. To this end, the columns contained 3.1 cc enzyme bed, 1 cc commercial catalyst (1% Pt on carbon, Englehard) and 5 cc IRA-68 ion-exchange resin. The catalyst should convert the acetaldehyde to acetic acid, which is adsorbed by the ion-exchange resin. The enzyme bed was prepared by the titanium process.

The difference between the two columns was that the 61L column had each component layered onto each other, whereas the 61M column had the three components mixed together. The columns were run at 1.2 mL/min, to allow a similar contact time to the enzyme bed tests. The tests began on 6/25/90 and ran for a short (2 L/cc) duration. It became obvious that the amount of catalyst in the beds was not sufficient to oxidize the aldehyde. The columns were redesigned and the test repeated.

The new columns (69L and 69M) contained the same amount of enzyme bed, with 4 cc Pt catalyst and 5 cc IRA-68. A bed with Ag_2O as the catalyst rather than the Pt/C (layered) was also set up. All other conditions were kept the same as the first test. These columns were begun on 7/9/90 and ran for 4.5 L/cc. The silver based column showed no oxidation of the ethanol, and was shut down immediately. The layered column removed a greater amount of ethanol from the influent stream than the mixed column. Both columns were efficient at oxidizing the acetaldehyde to acetic acid, which was readily removed by the ion-exchange resin.

X. Column 62Pt was prepared by the titanium method on a support containing 1.1% Pt. This 6.1 cc column was run at 2.0

mL/min for 9 L/cc. The column oxidized 65% of the alcohol seen during the month and a half test. A similar column on 2.2% Pt support was run for 6 L/cc at 2 mL/min showed similar results. Both columns were prepared by the dilute titanium method. The initial duration of complete alcohol removal was 0.5 L/cc.

XI. A series of six columns were run to compare the effect of different concentrarions of Pt on the support, and also to test modifications in the immobilization process. The series was prepared by the titanium method with a 91 hour incubation period. The '71AW' set had a more detailed washing procedure for the beds at each stage of the process. The '71A-' set used much less wash, more in line with the published procedure. Both sets had 0.5% Pt, 1.1% Pt and 2.2% Pt supports. As expected, the results demonstrated that the 2.2% Pt bed with extra wash performed the most effective convertion. In each case, washed columns outperformed the less washed counterparts and higher platinum concentration columns functioned for a longer duration. The 71AW2 column was effective for 28 L/cc, before restricted flow caused its demise. The other columns were run for shorter duration depending on the ability to oxidize the ethanol challenge solution.

XII. A column (80PT20) was prepared to test the change from 1% hexanediamine to 1% ethylenediamine as a linkage agent in the immobilization process. The change had been successfully demonstrated on a urease column. The test was done with 1% Pt support and lasted for a duration of 8 L/cc at 2.0 mL/min flow rate. In addition, two columns (85A, 85B) of the same immobilized

batch were stacked with Pt/C. One had the commercial Englehard 1% catalyst, while the other column contained a material prepared by UMPQUA. All three columns contained roughly 5 cc of enzyme bed. The results show that despite good initial activity, the beds did not oxidize ethanol for a significant period on time. The latter tests were run for less than 5 L/cc before abortion.

XIII. A series of three columns (89A-C) was prepared on 2% Pt support with 1% ethylenediamine as the linkage agent, using a 96 hour incubation period. (All further columns described are prepared with ethylenediamine, the concentration of this reagent was gradually increased up to 3.5%). The first column containing 5 cc media was equipped with a 1% Pt/C bed and an IRA-150 bed, for complete organic removal. The other two columns contained 14 cc, and were tested with 20 PPMV and 200 PPMV ethanol respectively. There were some indications that the ability of the enzyme beds was being limited by the contact time of the bed. This test was designed to address this hypothesis and was started on 8/28/90.

The short column worked effectively for 11 L/cc, removing over 90% of the total organic carbon from the waste stream. The long column worked extremely well, removing over 80% of the ethanol for 21 L/cc, and is still in operation eight months after its initiation. The high concentration column did not work well, and was operated for only 1.8L/cc before closing down operation. The problem was that the concentration of ethanol was much greater than the amount of oxygen dissolved in the water stream. There was simply not enough oxygen present to handle this tenfold increase.

XIV. A column (94PtA) was prepared with a 4.2% Pt deposition on the support by the titanium method. This 5 cc column, started 9/6/90, ran at 2.0 mL/min for 21 L/cc. The column worked well, as efficient as the 89A column. It was determined that increasing the Pt concentration beyond 2% provided no added benefit.

Material from the same 4.2% batch (94B) was tested with 200 PPMV ethanol with similar results to the 89C test. Another column from this batch was tested with several different primary alcohols as described in the main text.

XV. An alcohol oxidase bed on 2% Pt prepared by the titanium method was mixed with a support that had DC-1500 quaternary ammonium salt deposited on the surface. The column (II-6E) contained 5.3 cc of enzyme bed, and was run at 2.0 mL/min flow rate. The column removed half the ethanol from the challenge solution for a duration of 5.8 L/cc. This column was one of several columns prepared with quaternary ammonium salts as part of the biocidal column studies. These salts were dried onto support both before and after the derivitization process, in conjunction with both enzymes. The II-6 column was the only bed in the set that was worth running beyond the initial 3 L/cc, based on enzyme performance and microbial activity checks.

XVI. Two elongated columns (II-14-1 and II-14-2) were prepared on 10/22/90 with 11.5 cc and 11.8 cc beds respectively. The columns were prepared on 2% Pt support by the titanium method and were operated at 2.0 mL/min flow rates. Both columns had a 20 cc Pt/C catalyst column and the II-14-2 column had a 50 cc ion-

exchange column containing both IRA-68 and IRN-78. The components of the ion exchange column were layered onto each other.

The columns were begun on 10/24/90, and run for 8 L/cc and 4 L/cc respectively. Both columns oxidized the ethanol completely during the first L/cc, then gradually fell off to 75% by 5 L/cc throughput. The amount of acetaldehyde produced by both columns was greater than expected with a catalyst bed. The tests were discontinued in lieu of other test beds.

XVII. An 8.9 cc enzyme bed on 2% Pt, was prepared by the titanium method (II-20). The column, started on 11/7/90, was equipped with a 1% Pt/C post-column bed to convert aldehydes to acids. At the flow rate of 2.0 mL/min, this column oxidized most of the organic carbon species to organic acid for 20 L/cc, and is still in operation. Data was collected both before and after the post column. The ethanol that passes the enzyme bed is not affected by the catalyst, but the acetaldehyde is decreased by a considerable amount. This column is described in the main text.

XVIII. A pair of columns were set up on 11/14/91 to test whether copper could replace platinum as the metal deposited onto the support. One 11.9 cc column contained 1% Cu deposited onto the support, while the other 10.5 cc had 2% Pt and served as a control. It also was used as a batch test. Another objective of the copper test was to determine if the metal could remove the peroxide product without causing the oxidation of acetaldehyde to acetic acid.

The columns were prepared by the titanium method with the

ethylenediamine concentration increased to 2%. The enzyme incubation period was 117 hours. The flow rate for each bed was 2.0 mL/min. The control column oxidized over 75% of the ethanol influent during the 14 L/cc trial. The copper column was not as efficient at ethanol removal through the 13 L/cc that it was run, but did leave a higher percentage of the products as acetaldehyde. The result is somewhat surprising in that Cu^{2+} is considered an inhibitor of the alcohol oxidase enzyme.[1]

XIX. The membrane reactor system was initially set into operation during November 1990. The first trial (II-23M) used a total loop volume of 30 mL. This solution consisted of 10 mL (10,000 EU) alcohol oxidase in sucrose solution, 160 mg (450,000 EU) catalase and 20 mL of distilled water. The closed loop was circulated at 1.3 mL/min. The open loop contained oxygen saturated 20 PPMV ethanol influent circulated on the outside of the membrane tubes. This solution could diffuse into the membrane, which was rated small enough (100,000 NMWC) to prevent the enzymes from diffusing out.

The influent solution was initially circulated at 1.3 mL/min, but was gradually increased to 1.7 mL/min during the course of the 3.5 liter test. Distilled water was circulated in a closed loop through the external system overnight, while the internal loop remained at constant flow. This likely had the effect of washing away the sucrose buffer, although no measurement of such was ever attempted. The results showed a gradual deactivation of the enzyme. Over the four days of testing, the efficiency of the

system decreased from 75% to 50%. The enzyme loop solution faded from red to peach color and became opaque during the course of the test. This did not appear to effect the results. Further testing of this system was justified.

The second membrane reactor (II-26) used the same membrane housing, which was flushed with dilute NaOH after the initial use. The internal loop was increased to 50 mL, with the difference in volume taken up by using 30 mL (30,000 EU) of the alcohol oxidase. The influent solution was increased to 100 PPMV oxygen saturated ethanol. This test, begun on 11/19/90, ran for 6 liters and was reasonably effective at oxidizing the higher concentration of ethanol. This column is also described in the main text.

XX. The high pressure columns involve a method for increasing the amount of oxygen dissolved in the influent stream. The first column (28-HP) contained a 14.8 cc media bed, prepared by the titanium method with an incubation period of 120 hours and flowed at 2.0 mL/min. The oxygen (30 PPMV) was fed into the stream via a gas/liquid saturator designed by UMPQUA research. System pressure was controlled by a needle valve and held constant at 40 PSIG.

Initially, the column removed all the ethanol in 100 PPMV and 200 PPMV influent solutions. At 300 PPMV, only a trace amount of ethanol remained. When the influent stream was increased to 500 PPMV, 70% of the ethanol was oxidized. These results are discussed in the main paper. The test was extended to 20 liters total throughput of 500 PPMV ethanol and showed a gradual decrease in the column efficiency.

A second high pressure column was prepared using similar conditions (II-35). The bed volume was 17.7 cc with a 2 mL/min flow rate. The column was operated with an internal pressure at 50 PSIG and had the oxygen pressure systematically varied throughout the test. The column began operating on 12/9/90, and ran for 22 liters throughput of 500 PPMV ethanol. The concentration was then increased to 1000 PPMV, and the column operated for another 8 liters. The results of this test have been discussed in the main body of the report.

XXI. A test column prepared by the titanium method and incubated with enzyme for 90 hours was tested with a mixed alcohol ersatz solution. The composition of the influent was 20 PPMV ethanol, 10 PPMV methanol and 5 PPMV isopropanol. The column (II-33) was begun on 12/11/90, contained 13.9 cc of enzyme media and flowed for 5.6 L/cc at a rate of 2.0 mL/min. A Pt/C column was added after 1.5 L/cc in order to oxidize the residual aldehydes to organic acids. The post column also served to oxidize the isopropanol to acetone, much more efficiently than the enzyme bed. Isopropanol is <u>not</u> a primary alcohol and hence is also not a very good enzyme substrate.

XXII. A very long column was prepared to test whether the contact time was the key parameter for the enzyme function. This column was started on 12/15/90, with a 35.3 cc bed volume and a flow rate of 2.0 mL/min. The influent contained the ersatz solution described above. The column ran for 2.3 L/cc, much shorter than expected. The bed failed due to build-up of back-

pressure within the system. The higher length/diameter ratio used did not significantly change the product distribution, and the column began to allow ethanol through after 60 liters (1.8 L/cc). The column also did not increase the convertion of isopropanol to acetone beyond the results given by the shorter column.

XXIII. An enzyme bed was constructed using 2-methyl-1,5-pentanediamine (Dytek "A"), a liquid branched diamine as a linkage agent instead of ethylenediamine (II-41A). This reagent was added as a 10% solution in CCl_4, during the titanium derivatization process. A control column with 3.5% ethylenediamine in CCl_4 was prepared at the same time (II-41D). The incubation time was 120 hours. The columns were started on 1/10/91, with 20 PPMV ethanol influent at 2.0 mL/min flow rate. A duplicate pair of columns was stored at 4°C.

The Dytek column ran for 5 L/cc and was less effective at converting ethyl alcohol to acetaldehyde. By the end of the test, the Dytek column operated at 65% convertion, whereas the control column was at 75% conversion after 12 L/cc throughput. One possible reason for the difference is that two configurations are possible for the diamine, with the methyl group closer to either the support or the enzyme. The latter configuration may hinder the ability of the amine function to form the Schiff base bond with the glutaraldehyde, making the immobilization less efficient.

XXIV. A pair of columns were prepared to determine the effect of changes of pH and conductivity of the influent solution on the performance of the alcohol oxidase beds. These beds were prepared

using the titanium method with 3.5% ethylenediamine on 2% Pt support. Enzyme incubation was for 96 hours. The columns were initiated on 2/10/91 with 20 PPMV ethanol at 2.0 mL/min flow. After the columns were successfully run for 0.5 L/cc, the pH column influent was adjusted to pH=4.5 using HCl and the conductivity column set at K=90 with NaCl. Both columns performance gradually decreased during the period of operation under the modified conditions. After 8 L/cc throughput, the pH was increased to pH=5.5 and the conductivity restored to the original 0.9 value. The changes did not restore the column performance. The pH column broke down, but the conductivity column did maintained at the level where the change occured.

XXV. A full scale alcohol oxidase unibed was constructed and operation was begun on 2/14/91. The enzyme sub-bed contained 190 cc of media. The bed construction is given in full detail in Appendix B. The unibed was run at 25 mL/min flow with a 20-10-2 ersatz alcohol solution (ethanol/methanol/isopropanol). This column is described in the main text. After initially allowing ethanol and acetaldehyde through the bed during the first 140 L, the column settled out to removing 95% of the two carbon compounds through the next 300 L. The total TOC of the ersatz has been reduced from 15.1 mg/L to less than 2 mg/L. The bed oxidizes some of the isopropanol to acetone, but does not remove these three carbon compounds.

XXVI. A column was prepared with 2% palladium deposited on the support in place of platinum (II-53Pd) and compared to a 2% platinum column. Each bed, started on 3/12/91, contained 12.3 cc

media and was operated at 2.0 mL/min. Initially the 20 ppmv ethanol influent was oxidized completely in both columns, but the palladium column fell off rapidly in oxidizability. After 3 L/cc, the Pd column converted less than half of the ethanol to acetaldehyde, while the Pt control oxidized 75% of the ethanol after 5 L/cc.

XXVII. A column containing a 15.7 cc enzyme bed (II-33B) was started 3/7/91 after spending 3.5 months in storage at 4°C. At a 2.0 mL flow rate, this column oxidizes 85% of the 20 PPMV ethanol influent to acetaldehyde for 4.7 L/cc. This performance is similar to a column from the same prepared batch tested immediately after preparation, demonstrating that these enzyme beds can be stored cold for a short period of time.

XXVIII. A 7.5 cc enzyme bed (II-56) prepared by the titanium method on 2% Pt support was challenged with 20 PPMV formaldehyde, at 2.0 mL/min. The enzyme incubation period was 170 hours. This column, started on 4/8/91, oxidized the formaldehyde to formic acid for 10 liters of throughput. The column rapidly degraded through the next 15 liters of throughput and was oxidizing only 40% of the contaminant when the test was ended.

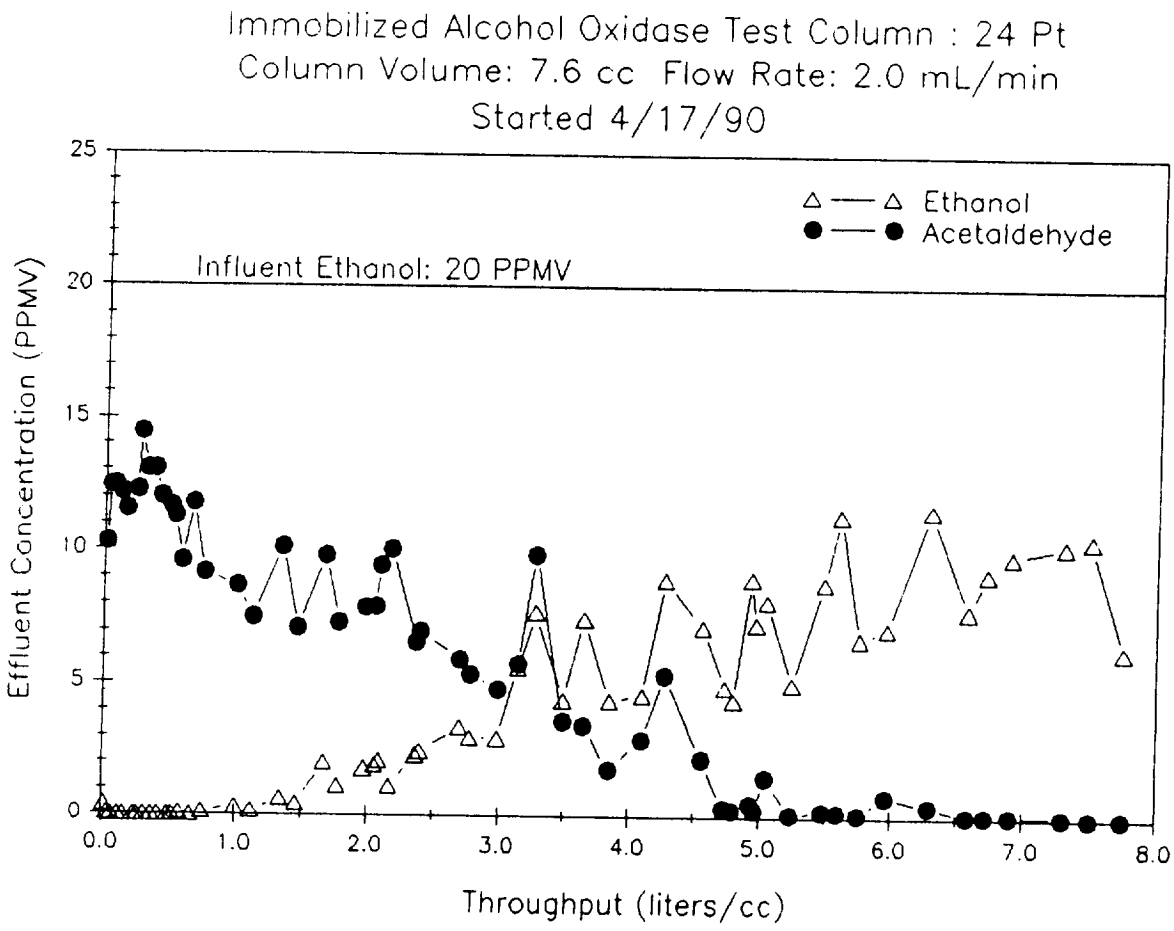
XXIX. A pair of 30 cc alcohol oxidase beds were prepared by the titanium process on 2% Pt support. The columns were packed into 1" polycarbonate columns rather than the typical 10 mm glass column. One of the two columns was subjected to gamma irradiation, while the other was to serve as a control. The experiment was designed to test whether a gamma ray treatment designed to

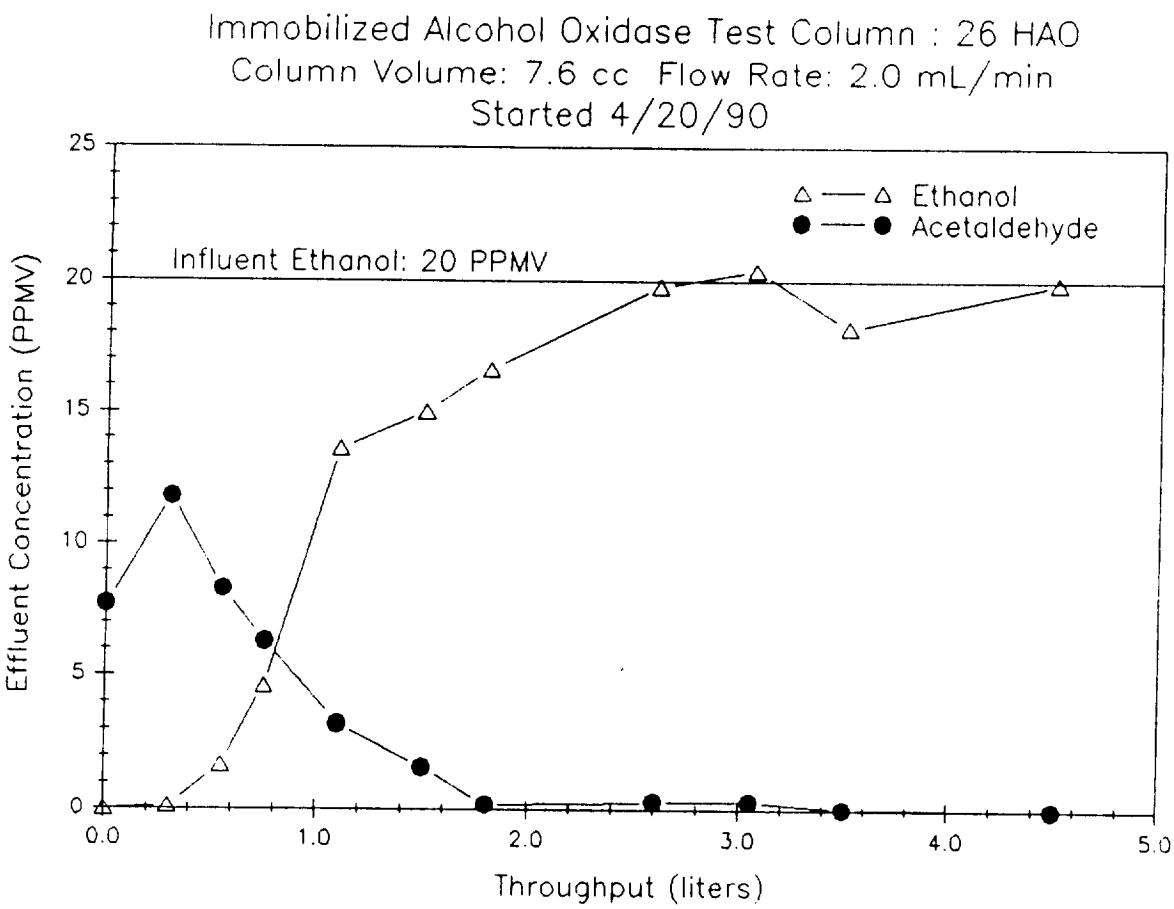
eradicate microbial growth would effect the performance of the enzyme.

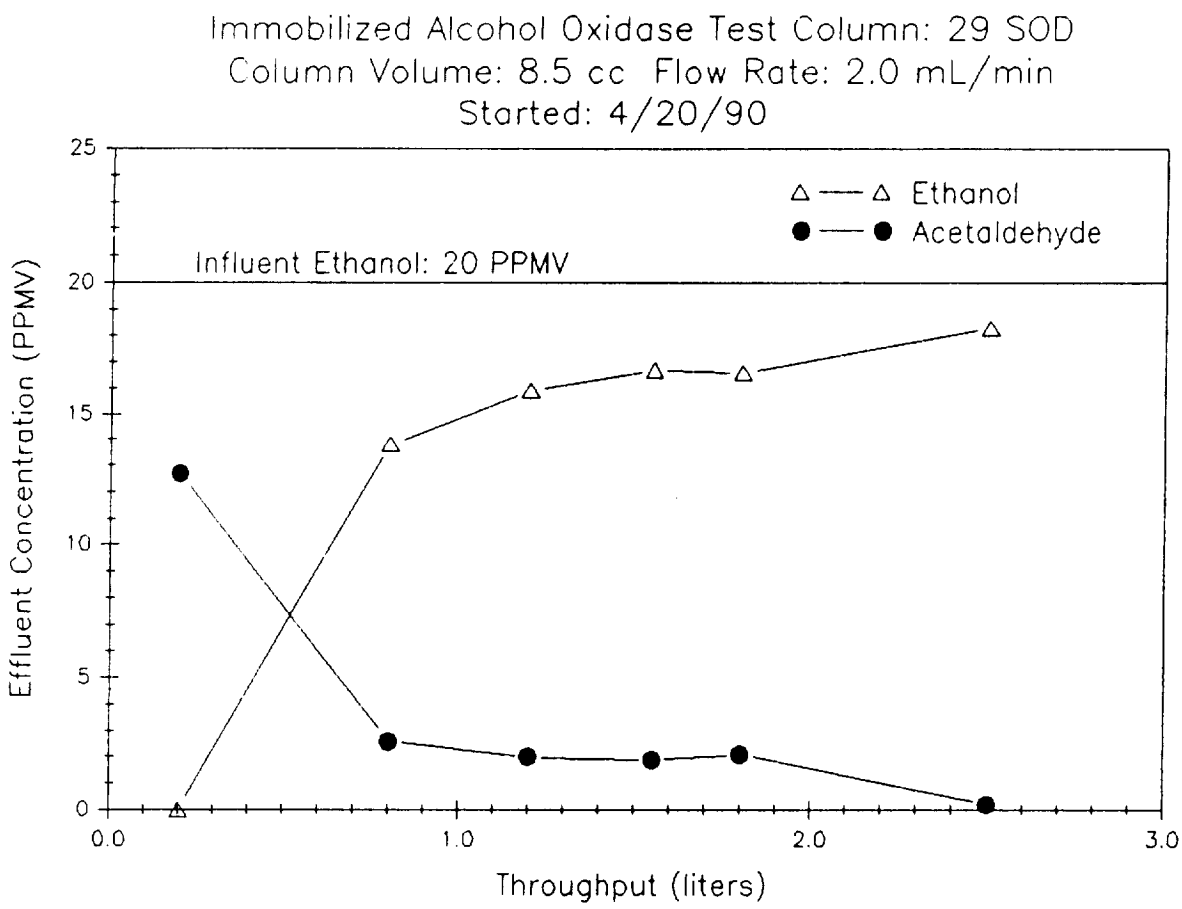
The control column did not return with the gamma ray column. The irradiated column (II-57A) was set up with 20 PPMV ethanol influent flowing through at 5 mL/min. The testing, started 5/2/91, showed that the initial performance of the enzyme bed was not effected by the radiation. A microbial sample of the intersticial water in the system after the treatment showed no microbes in the column. This test will continue to determine whether the lifetime of the enzyme bed will be affected.

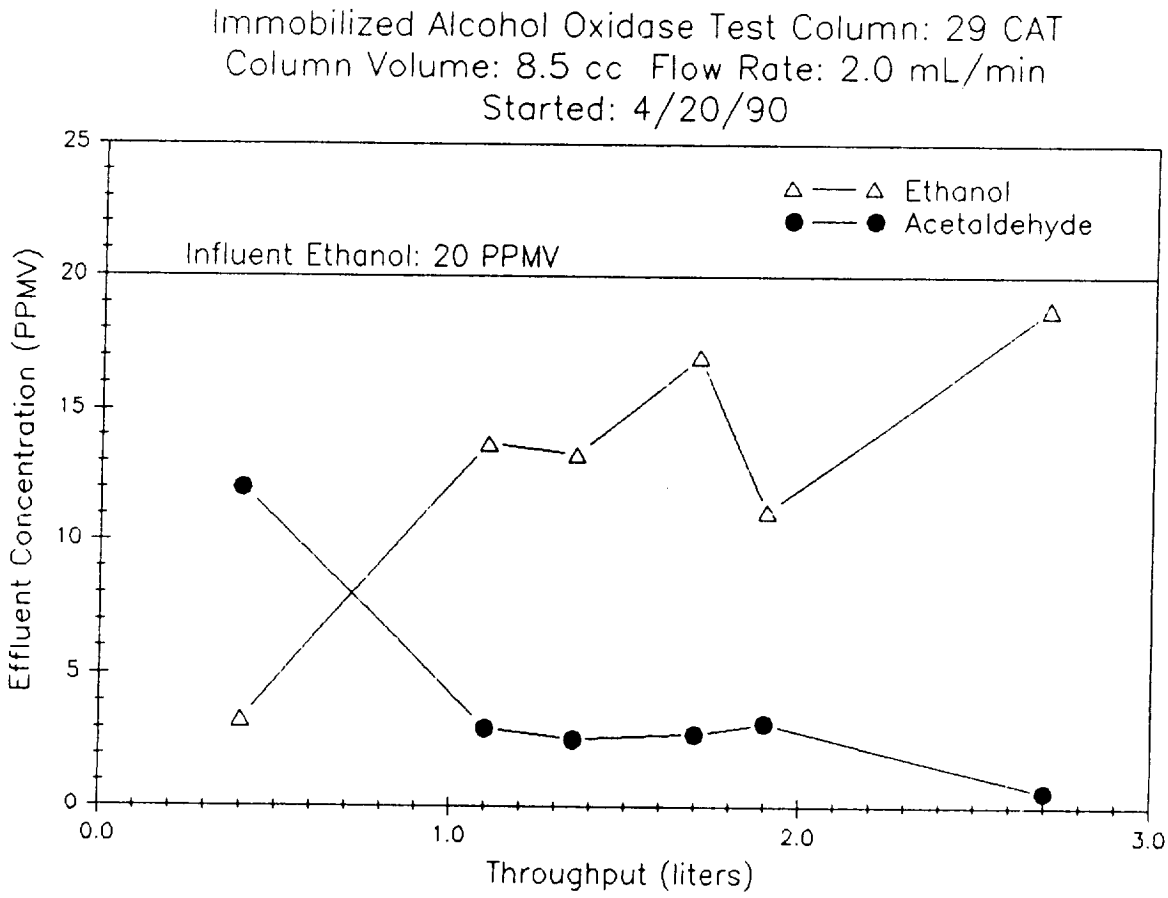
XXX. A pair of small columns were set up to determine the effect of elevated temperature on the immobilized alcohol oxidase. Each column contained 10 cc media and were flowed at 2.0 mL/min. The column tested at 45°C was immersed in a water bath to maintain temperature, while the control was run at room temperature in air. The elevated temperature column was deactivated shortly after the test was begun. After 4 liters of throughput, very little acetaldehyde was produced, whereas the control column was typical of previous enzyme beds.

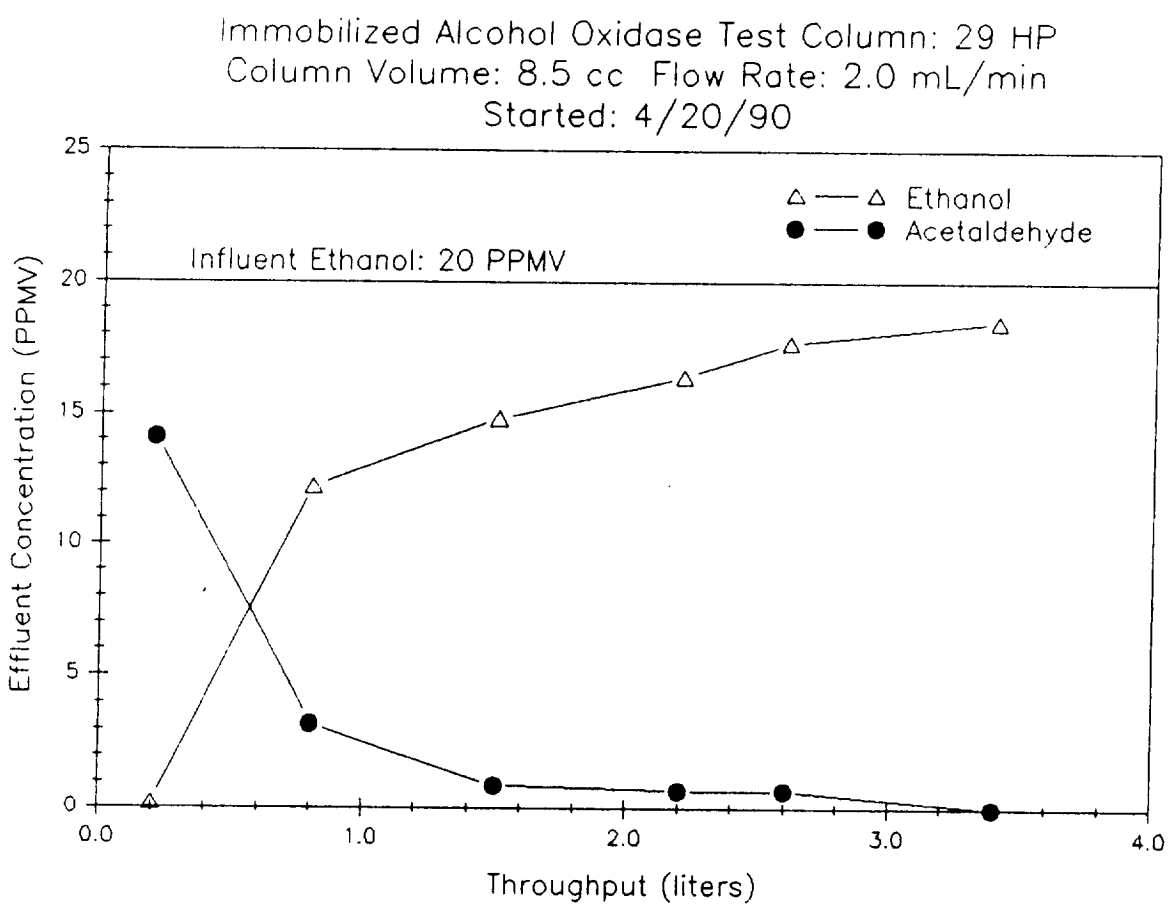
1. Hopkins, T.R. and Muller, F. "Biochemistry of Alcohol Oxidase" Biotechnology Division of Research and Development, Phillips Petroleum Company, Bartlesville, OK. 1982 Source Journal Unknown at this time.

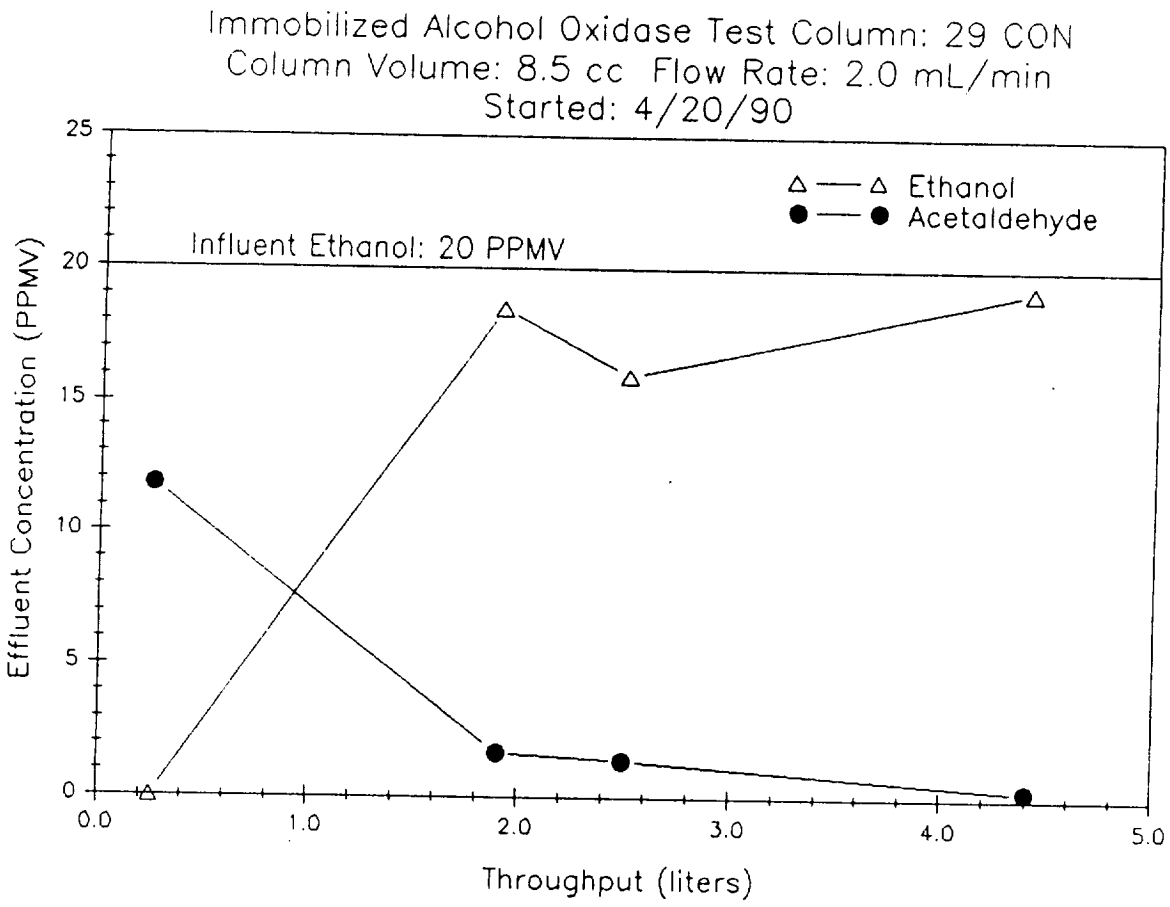

Immobilized Alcohol Oxidase Test Column : 24 Pt
Column Volume: 7.6 cc Flow Rate: 2.0 mL/min
Started 4/17/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0

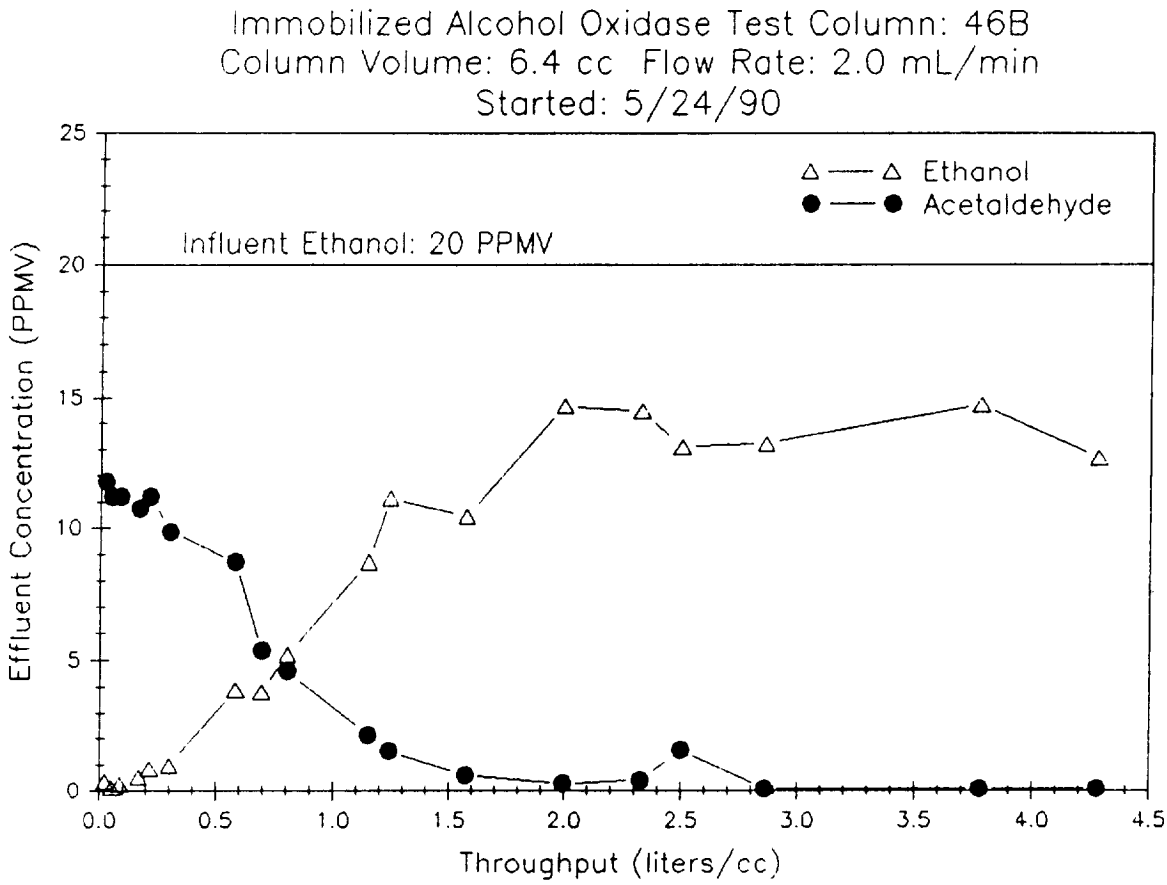

Immobilized Alcohol Oxidase Test Column : 26 HAO
Column Volume: 7.6 cc Flow Rate: 2.0 mL/min
Started 4/20/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters)
0
5
10
15
20
25
0.0
1.0
2.0
3.0
4.0
5.0

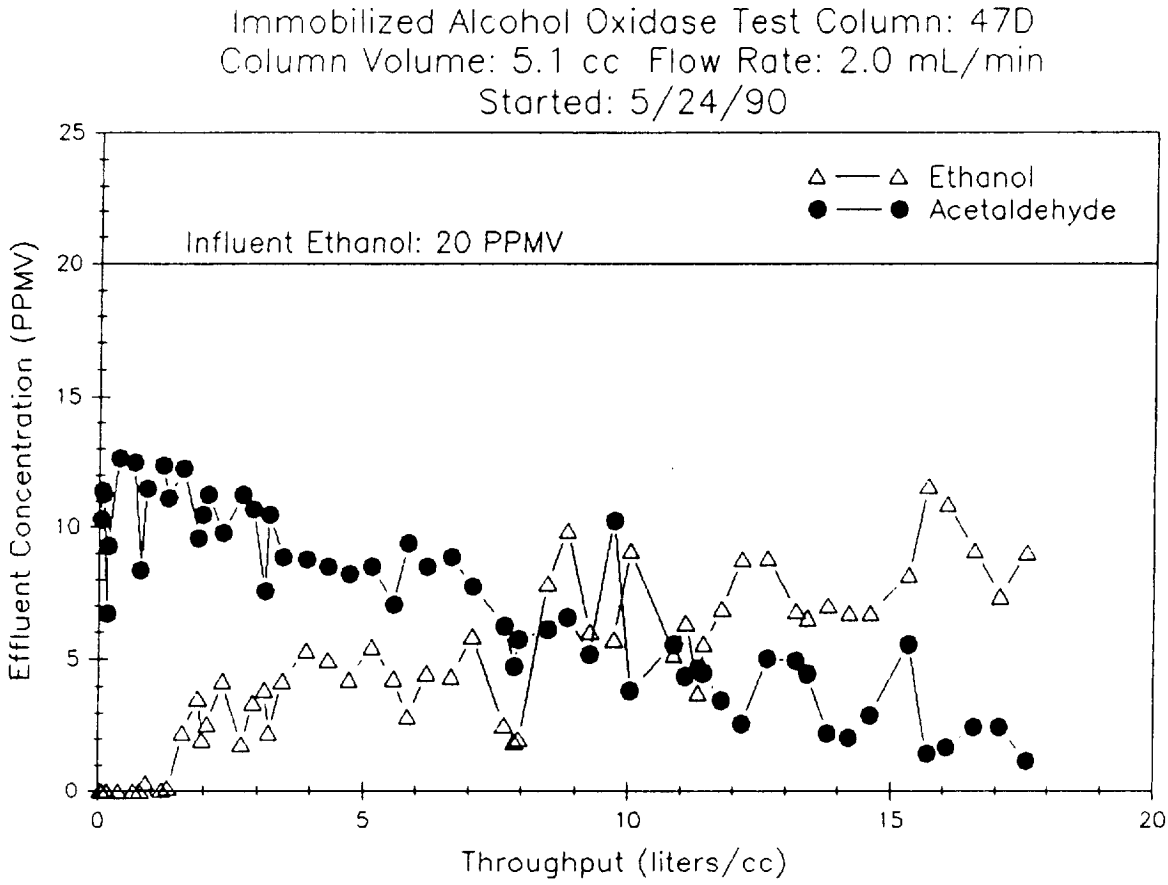

Immobilized Alcohol Oxidase Test Column: 29 SOD
Column Volume: 8.5 cc Flow Rate: 2.0 mL/min
Started: 4/20/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters)
0
5
10
15
20
25
0.0
1.0
2.0
3.0

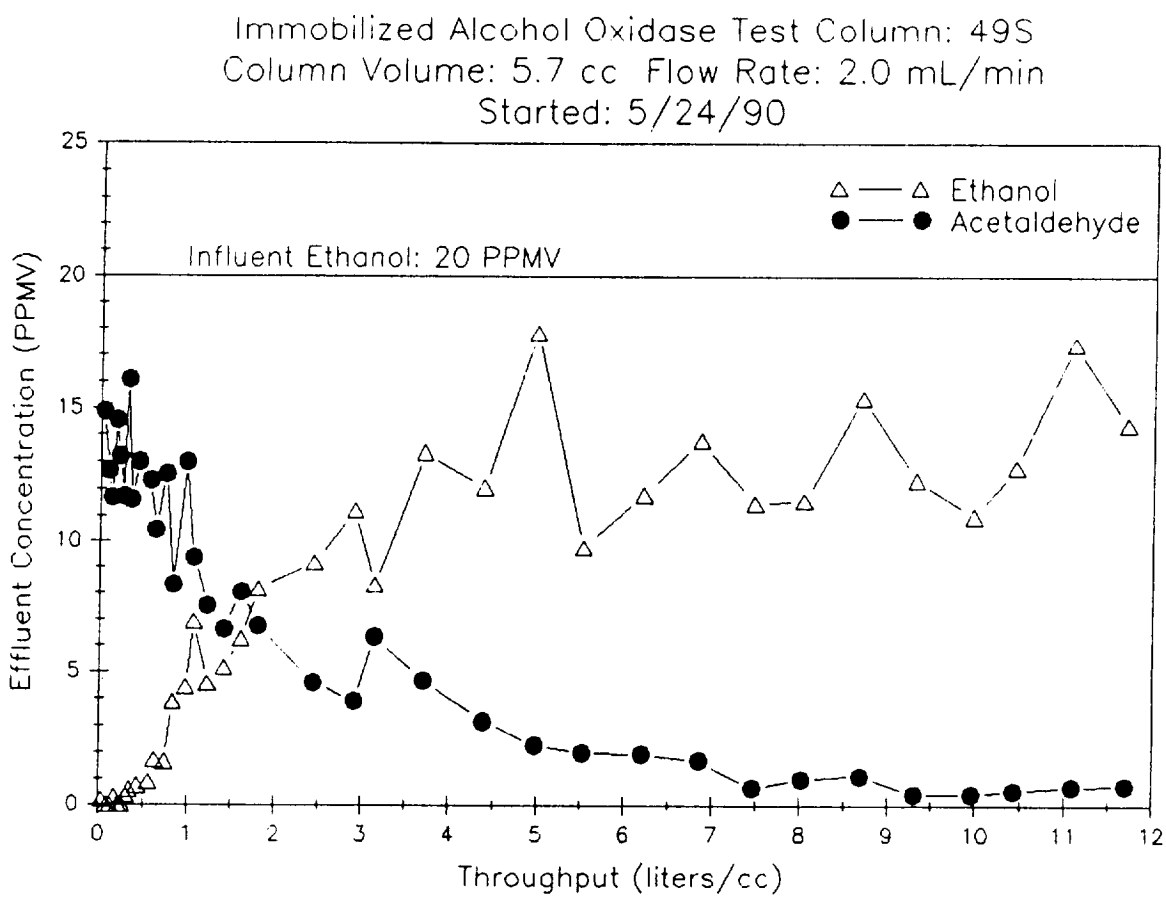

Immobilized Alcohol Oxidase Test Column: 29 CAT
Column Volume: 8.5 cc Flow Rate: 2.0 mL/min
Started: 4/20/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
25
20
15
10
5
0
0.0
1.0
2.0
3.0
Throughput (liters)

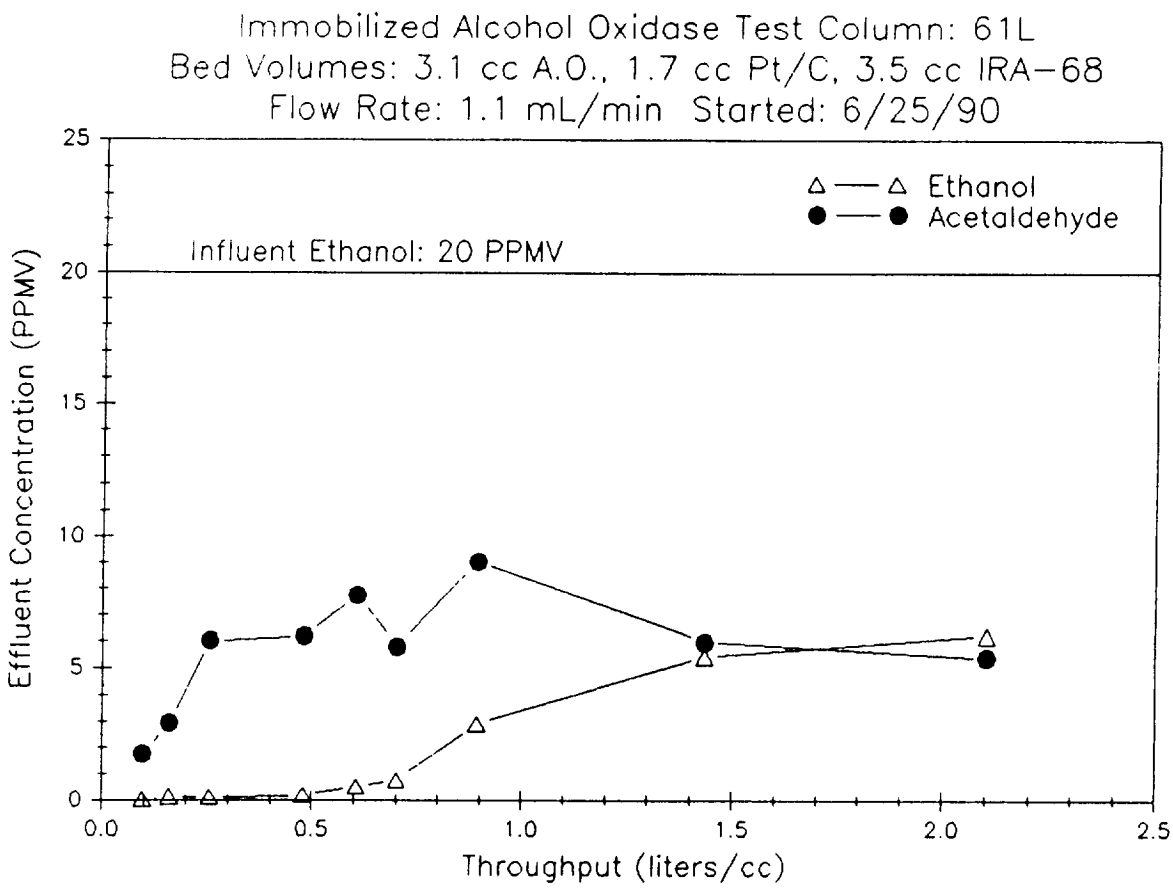

Immobilized Alcohol Oxidase Test Column: 29 HP
Column Volume: 8.5 cc Flow Rate: 2.0 mL/min
Started: 4/20/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
25
20
15
10
5
0
0.0
1.0
2.0
3.0
4.0
Throughput (liters)

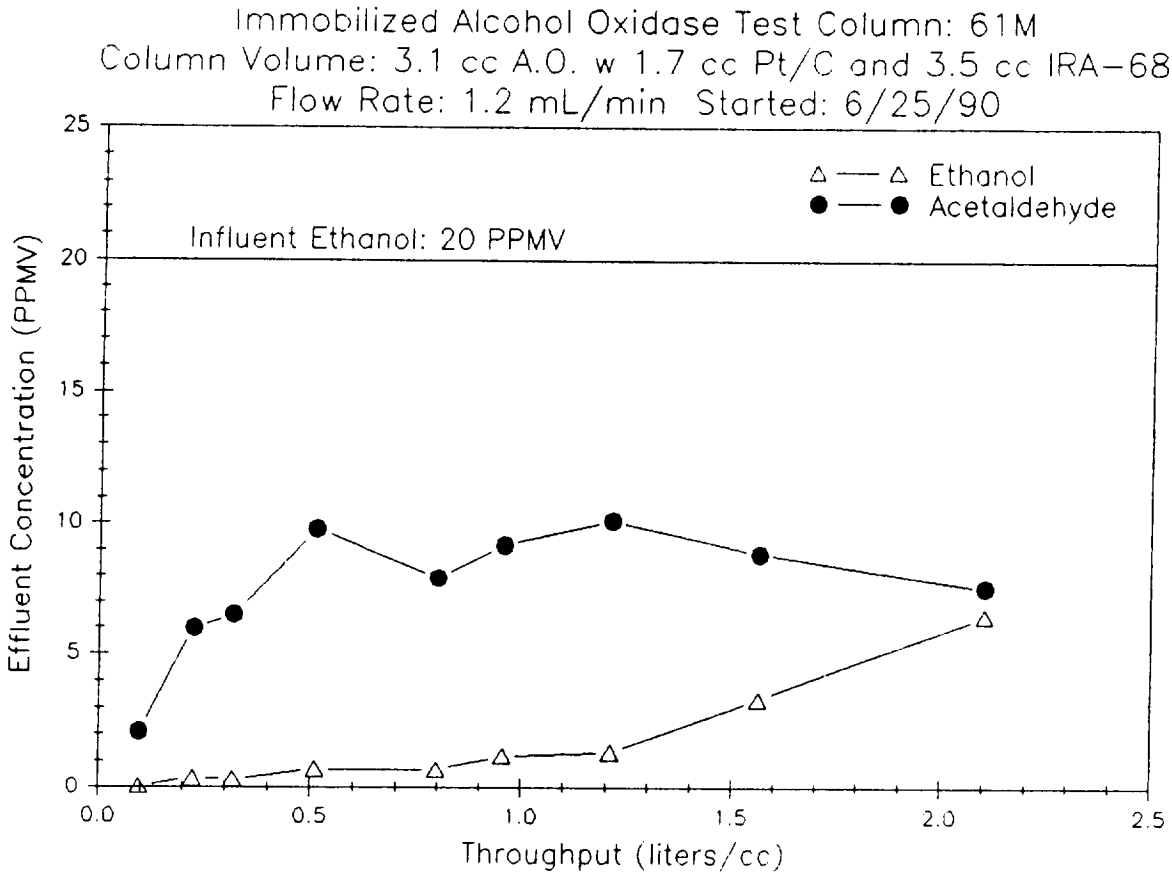

Immobilized Alcohol Oxidase Test Column: 29 CON
Column Volume: 8.5 cc Flow Rate: 2.0 mL/min
Started: 4/20/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
25
20
15
10
5
0
0.0
1.0
2.0
3.0
4.0
5.0
Throughput (liters)

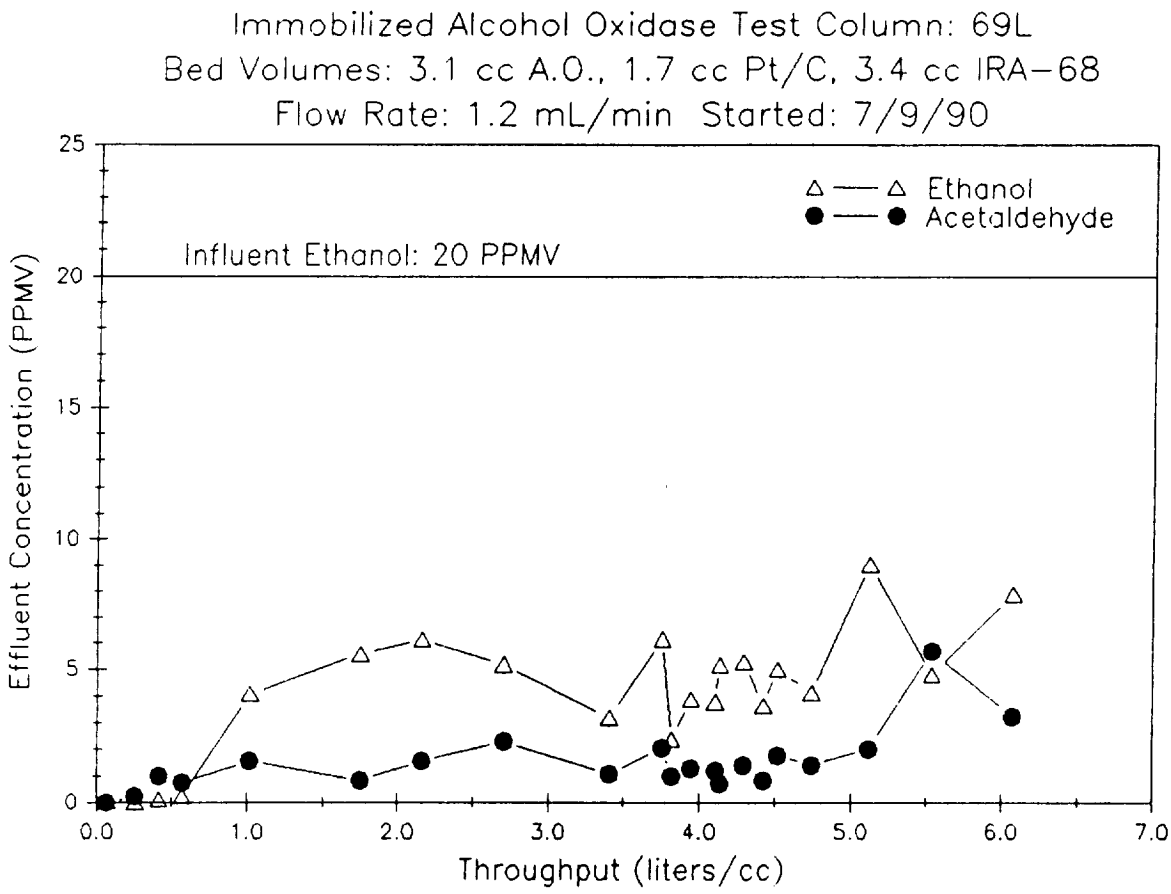

Immobilized Alcohol Oxidase Test Column: 46B
Column Volume: 6.4 cc Flow Rate: 2.0 mL/min
Started: 5/24/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5

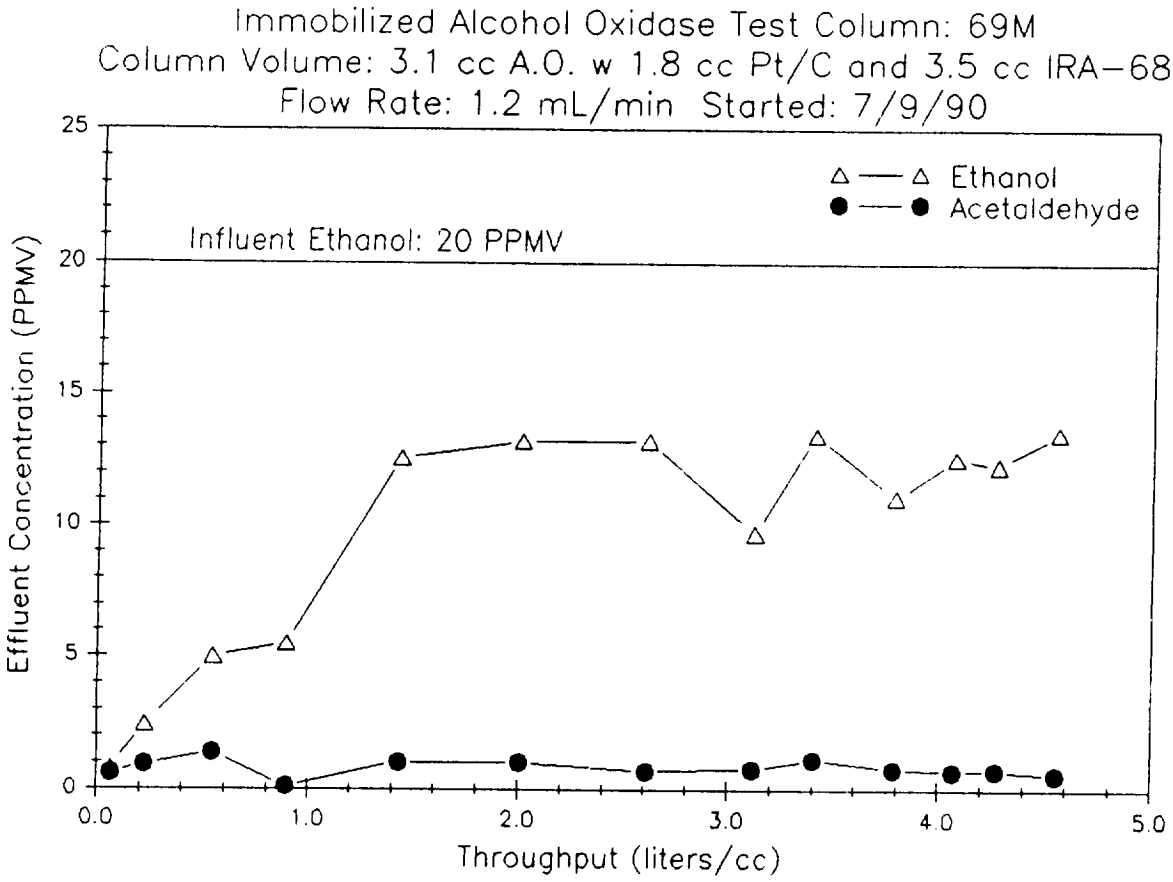

Immobilized Alcohol Oxidase Test Column: 47D
Column Volume: 5.1 cc Flow Rate: 2.0 mL/min
Started: 5/24/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25

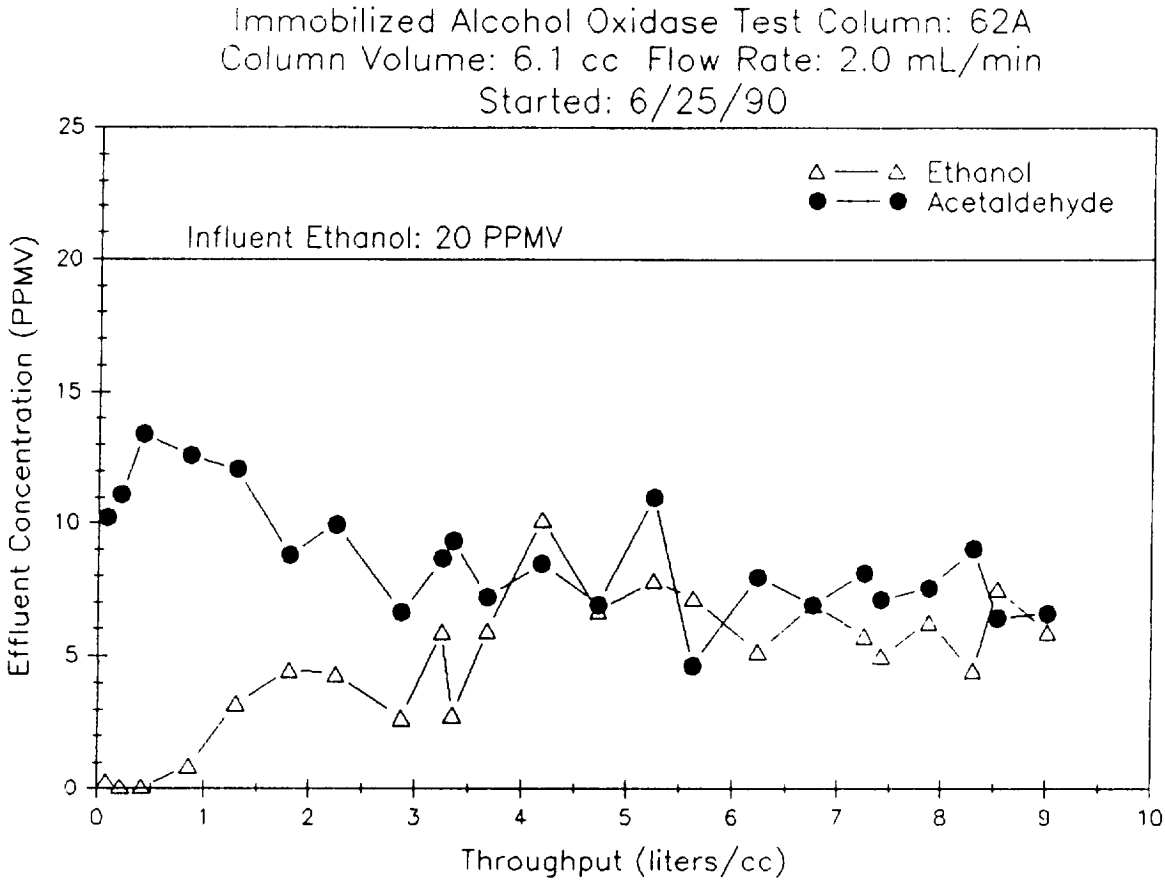

Immobilized Alcohol Oxidase Test Column: 49S
Column Volume: 5.7 cc Flow Rate: 2.0 mL/min
Started: 5/24/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0
1
2
3
4
5
6
7
8
9
10
11
12

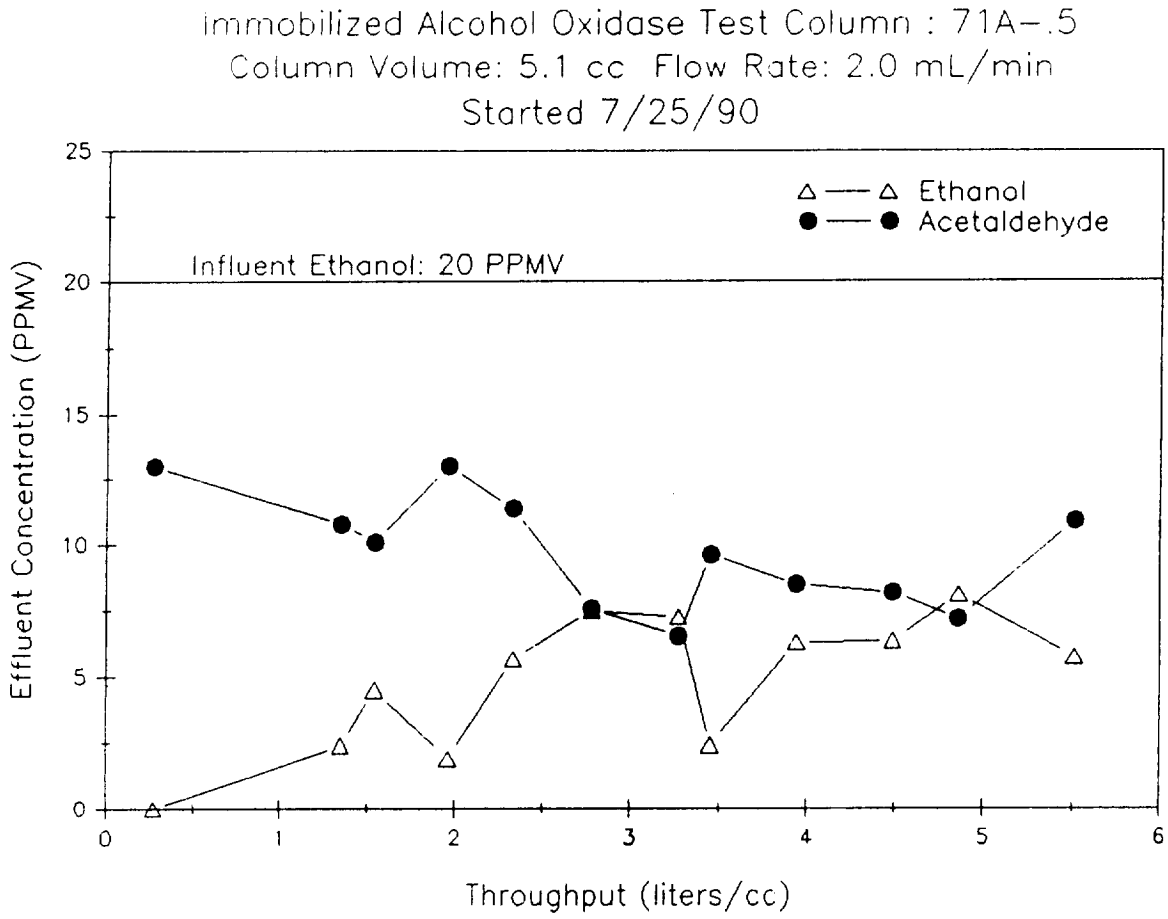

Immobilized Alcohol Oxidase Test Column: 61L
Bed Volumes: 3.1 cc A.O., 1.7 cc Pt/C, 3.5 cc IRA-68
Flow Rate: 1.1 mL/min Started: 6/25/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0.0
0.5
1.0
1.5
2.0
2.5

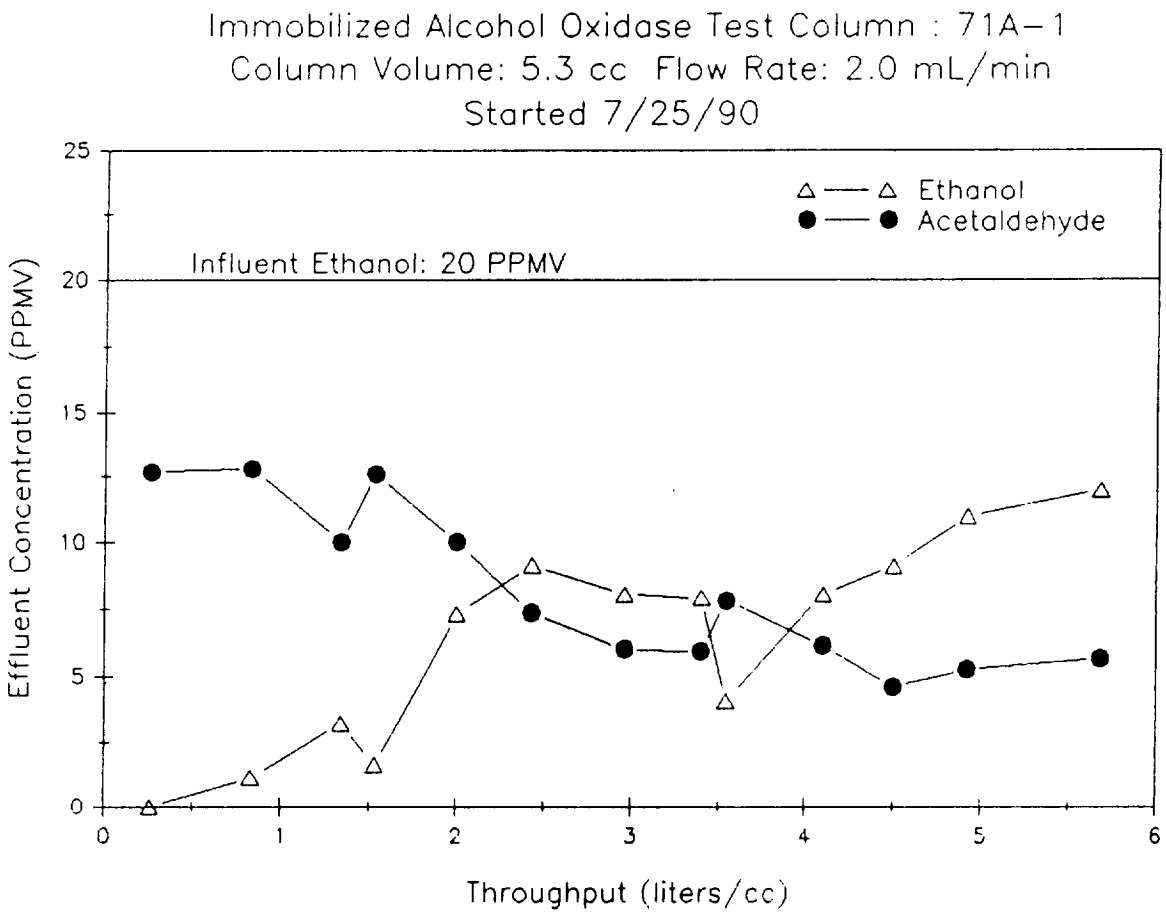

Immobilized Alcohol Oxidase Test Column: 61M
Column Volume: 3.1 cc A.O. w 1.7 cc Pt/C and 3.5 cc IRA-68
Flow Rate: 1.2 mL/min Started: 6/25/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0.0
0.5
1.0
1.5
2.0
2.5

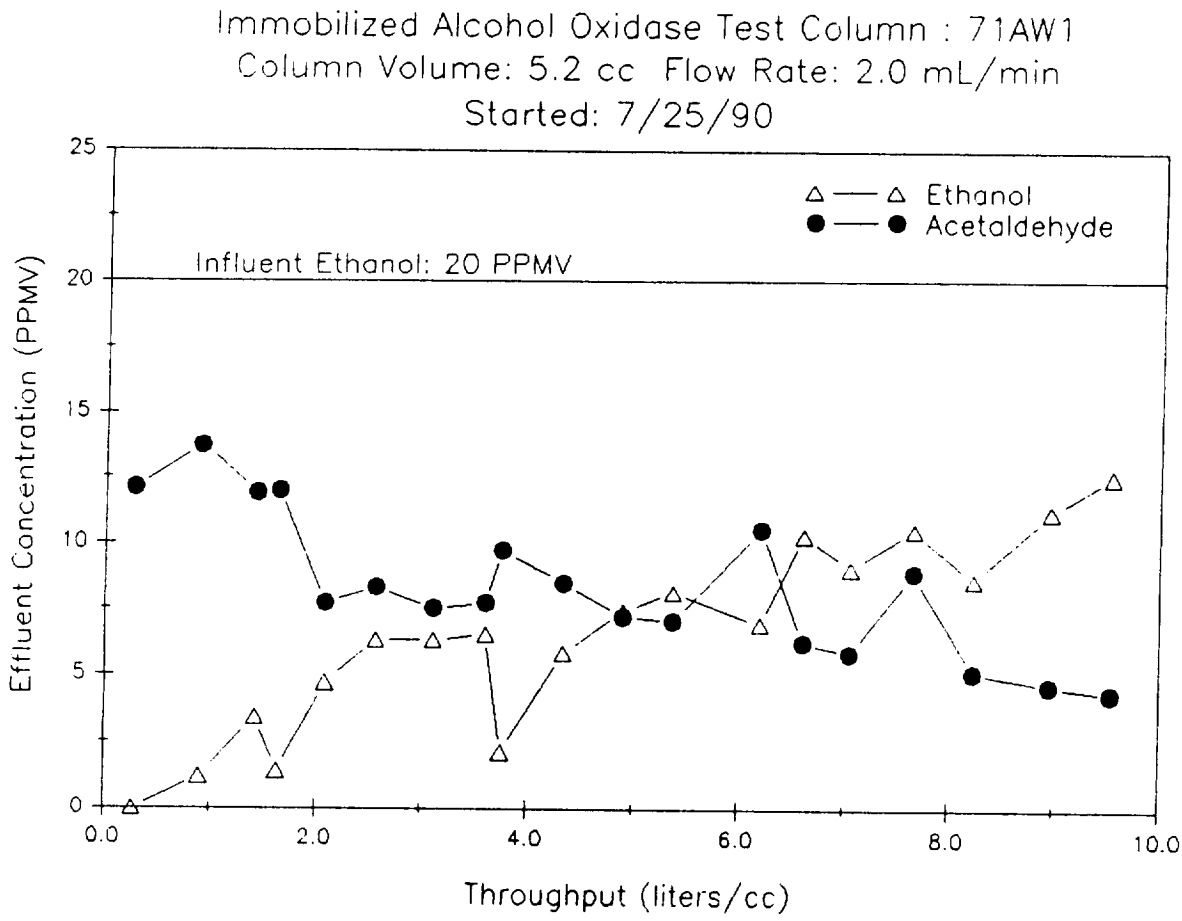

Immobilized Alcohol Oxidase Test Column: 69L
Bed Volumes: 3.1 cc A.O., 1.7 cc Pt/C, 3.4 cc IRA-68
Flow Rate: 1.2 mL/min Started: 7/9/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0


Immobilized Alcohol Oxidase Test Column: 69M
Column Volume: 3.1 cc A.O. w 1.8 cc Pt/C and 3.5 cc IRA-68
Flow Rate: 1.2 mL/min Started: 7/9/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0.0
1.0
2.0
3.0
4.0
5.0

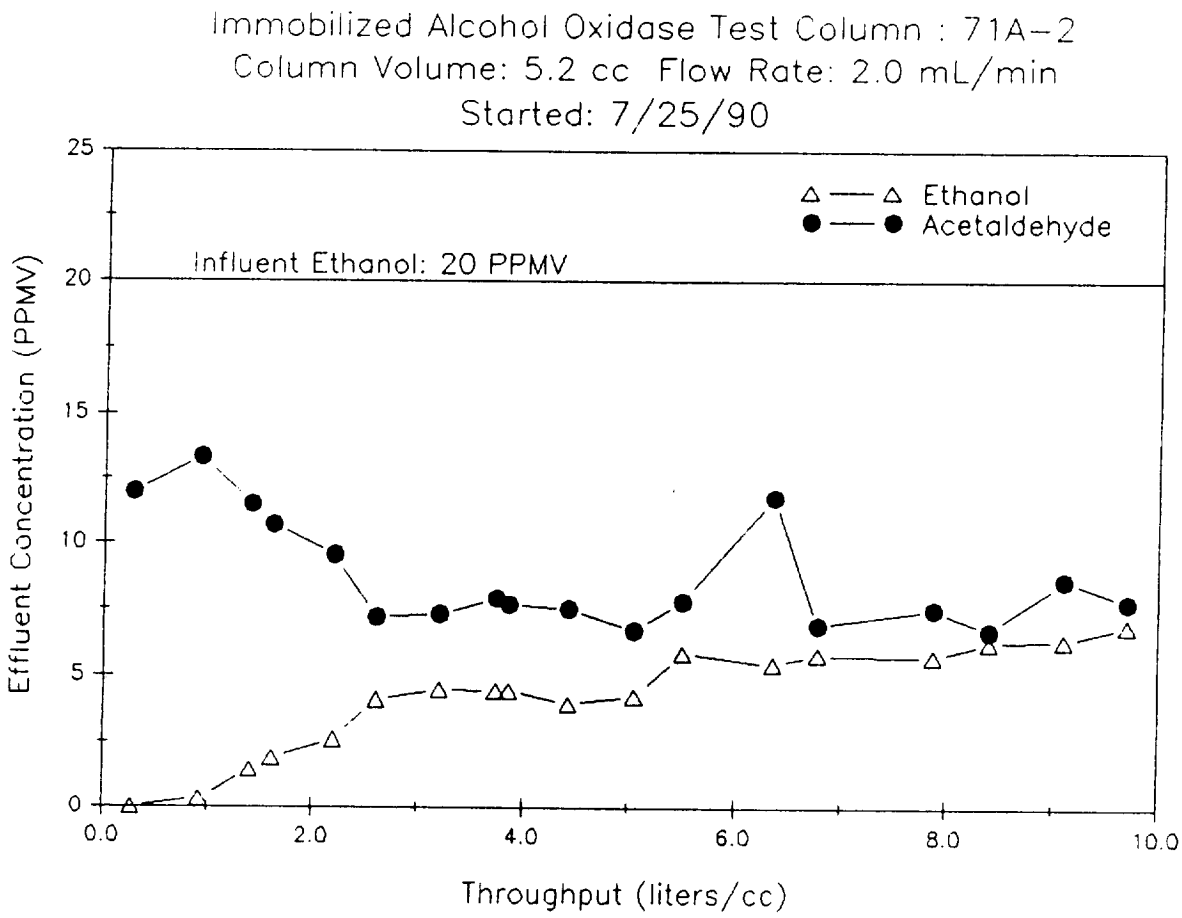

Immobilized Alcohol Oxidase Test Column: 62A
Column Volume: 6.1 cc Flow Rate: 2.0 mL/min
Started: 6/25/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0
1
2
3
4
5
6
7
8
9
10

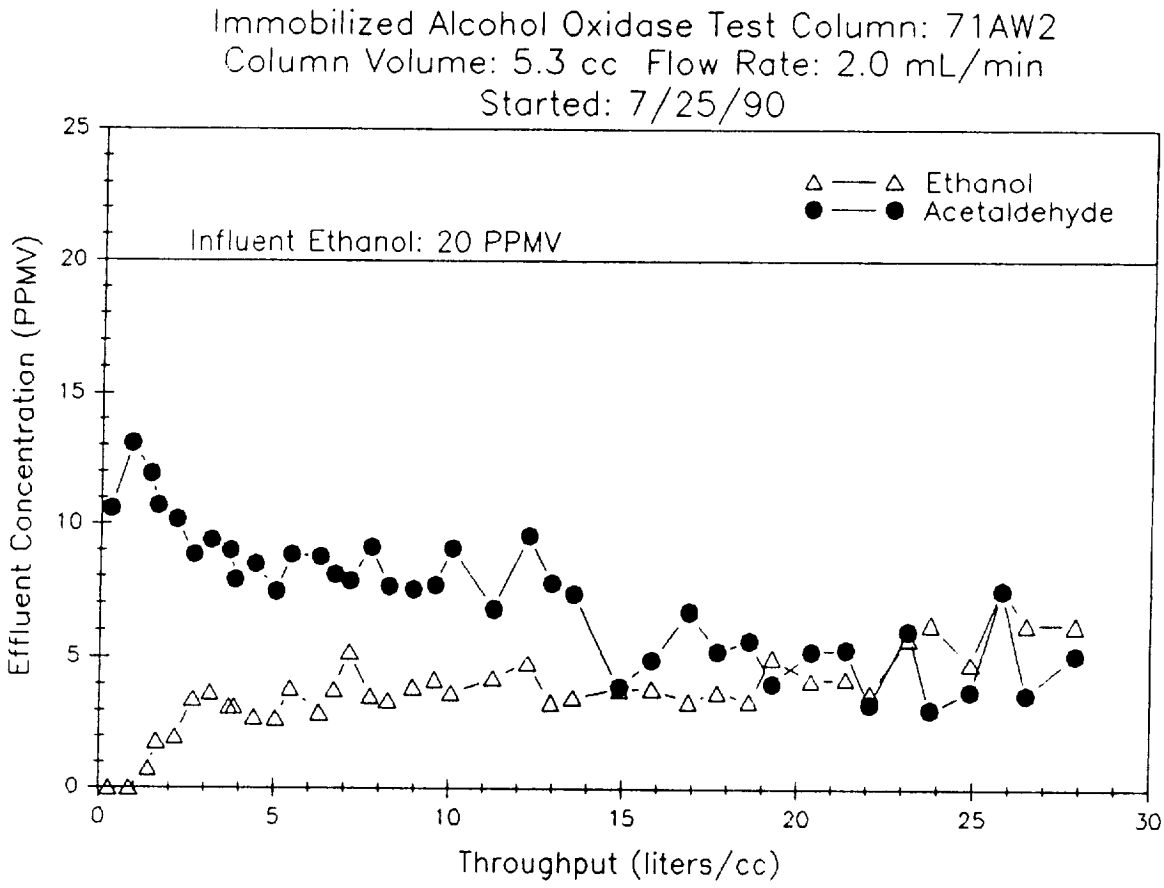

Immobilized Alcohol Oxidase Test Column : 71A-.5
Column Volume: 5.1 cc Flow Rate: 2.0 mL/min
Started 7/25/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0
1
2
3
4
5
6

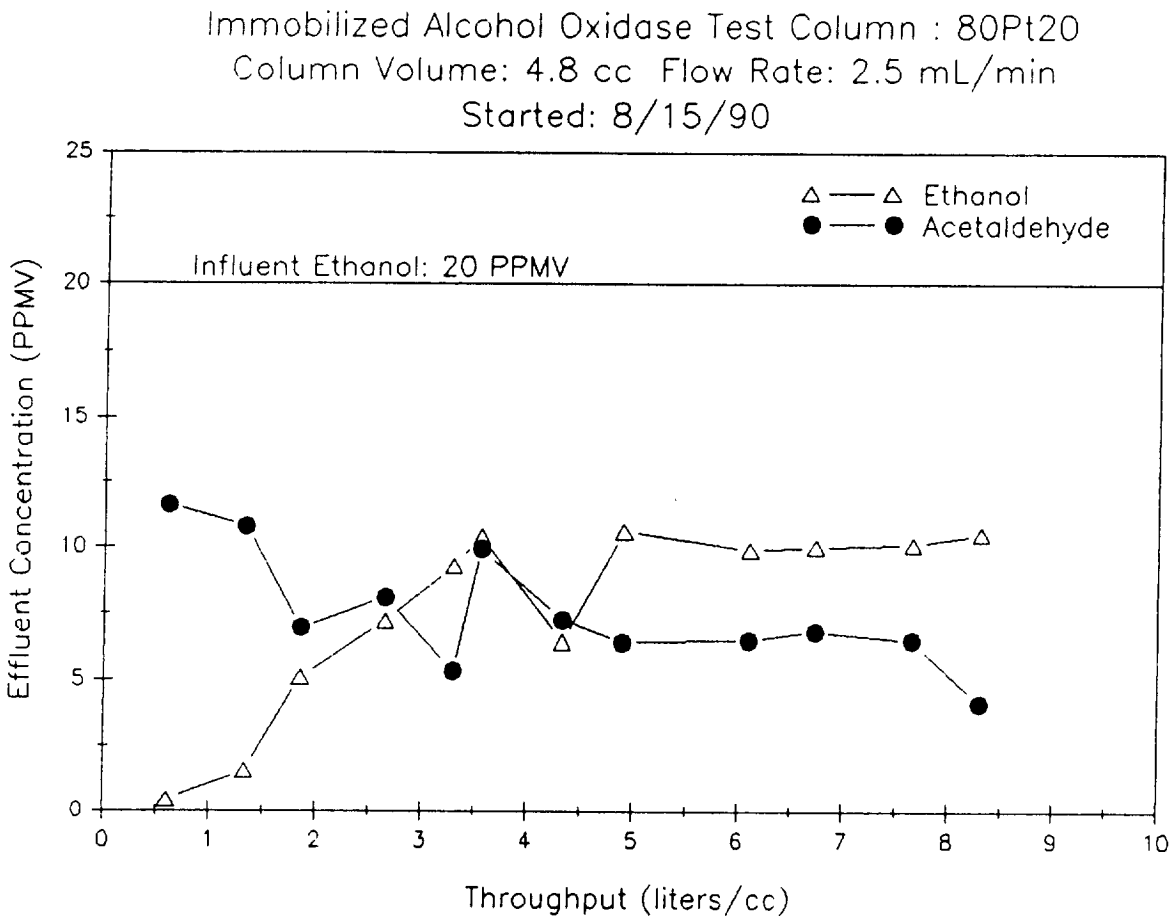

Immobilized Alcohol Oxidase Test Column : 71A-1
Column Volume: 5.3 cc Flow Rate: 2.0 mL/min
Started 7/25/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0
1
2
3
4
5
6

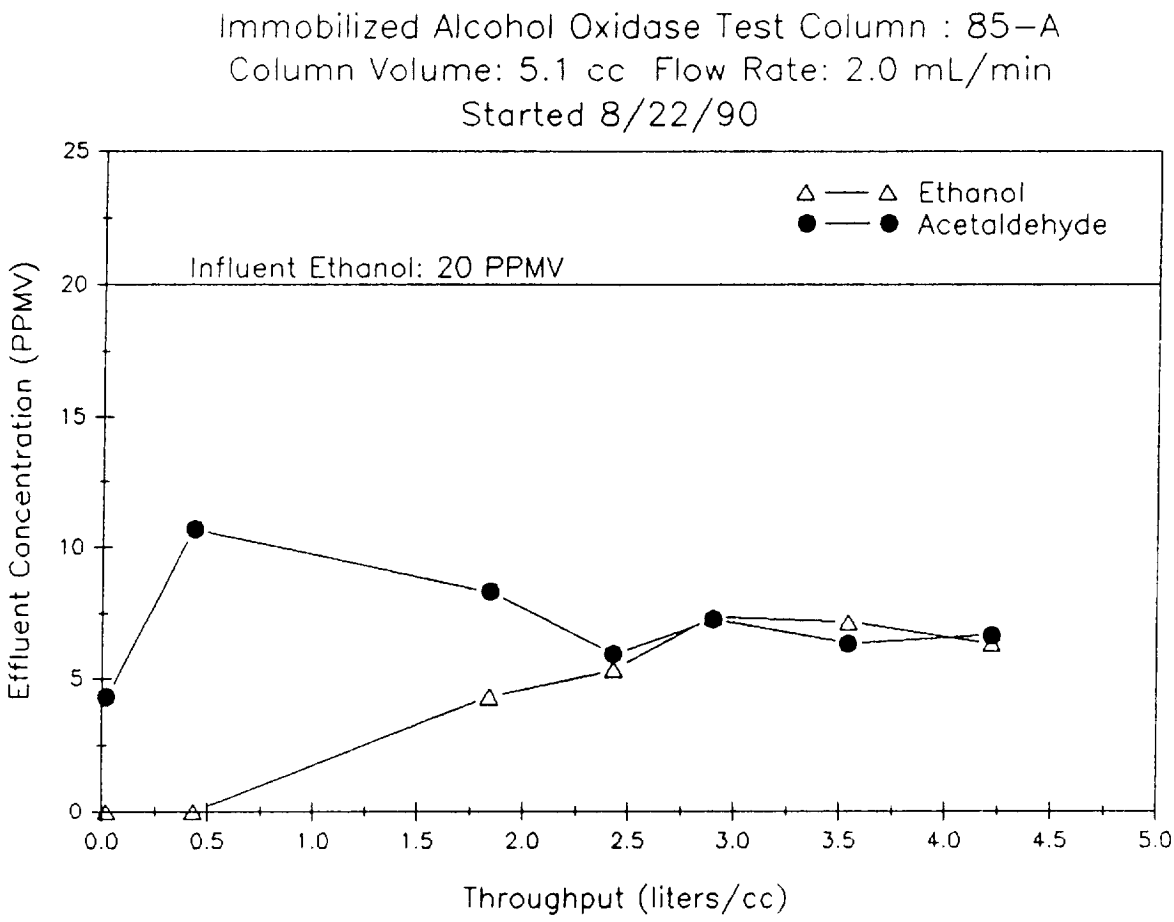

Immobilized Alcohol Oxidase Test Column : 71AW1
Column Volume: 5.2 cc Flow Rate: 2.0 mL/min
Started: 7/25/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0.0
2.0
4.0
6.0
8.0
10.0

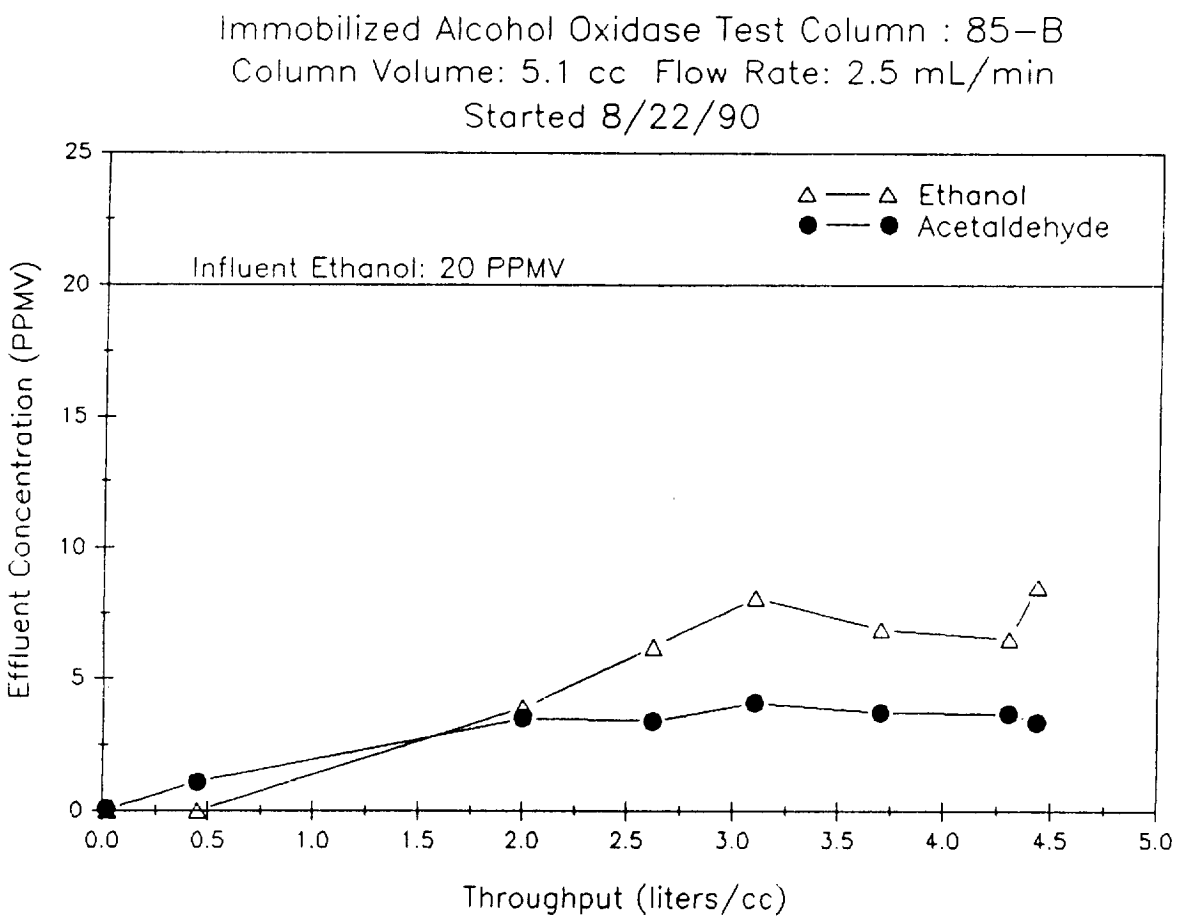
C-2

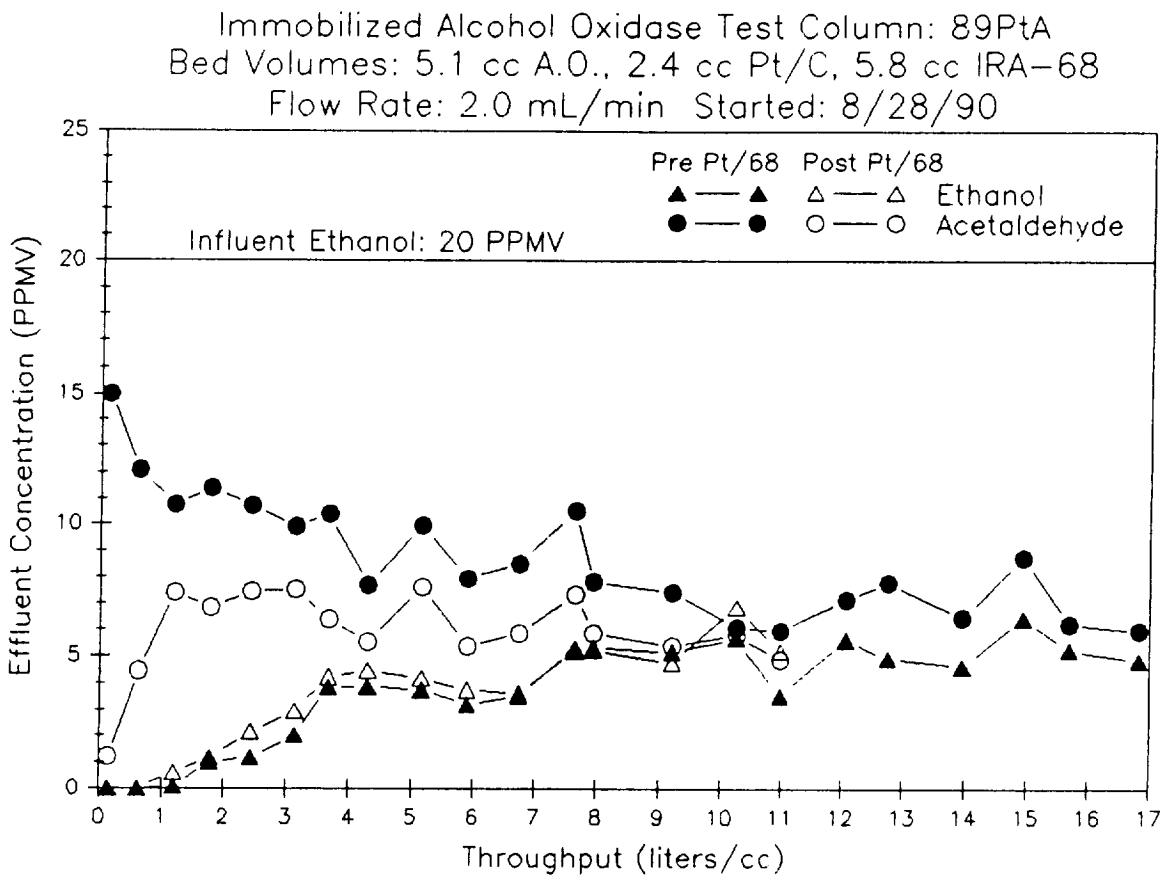

Immobilized Alcohol Oxidase Test Column : 71A-2
Column Volume: 5.2 cc Flow Rate: 2.0 mL/min
Started: 7/25/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
25
20
15
10
5
0
0.0
2.0
4.0
6.0
8.0
10.0
Throughput (liters/cc)

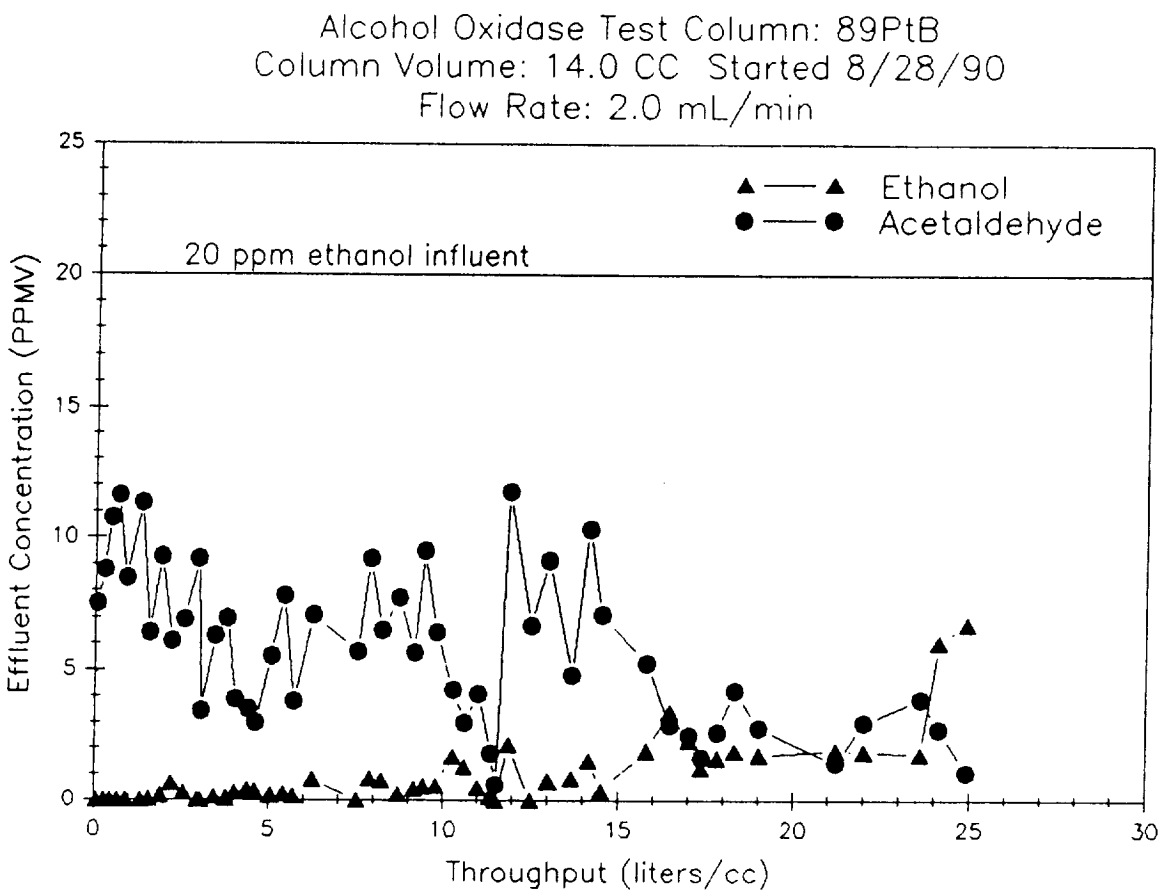

Immobilized Alcohol Oxidase Test Column: 71AW2
Column Volume: 5.3 cc Flow Rate: 2.0 mL/min
Started: 7/25/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
30

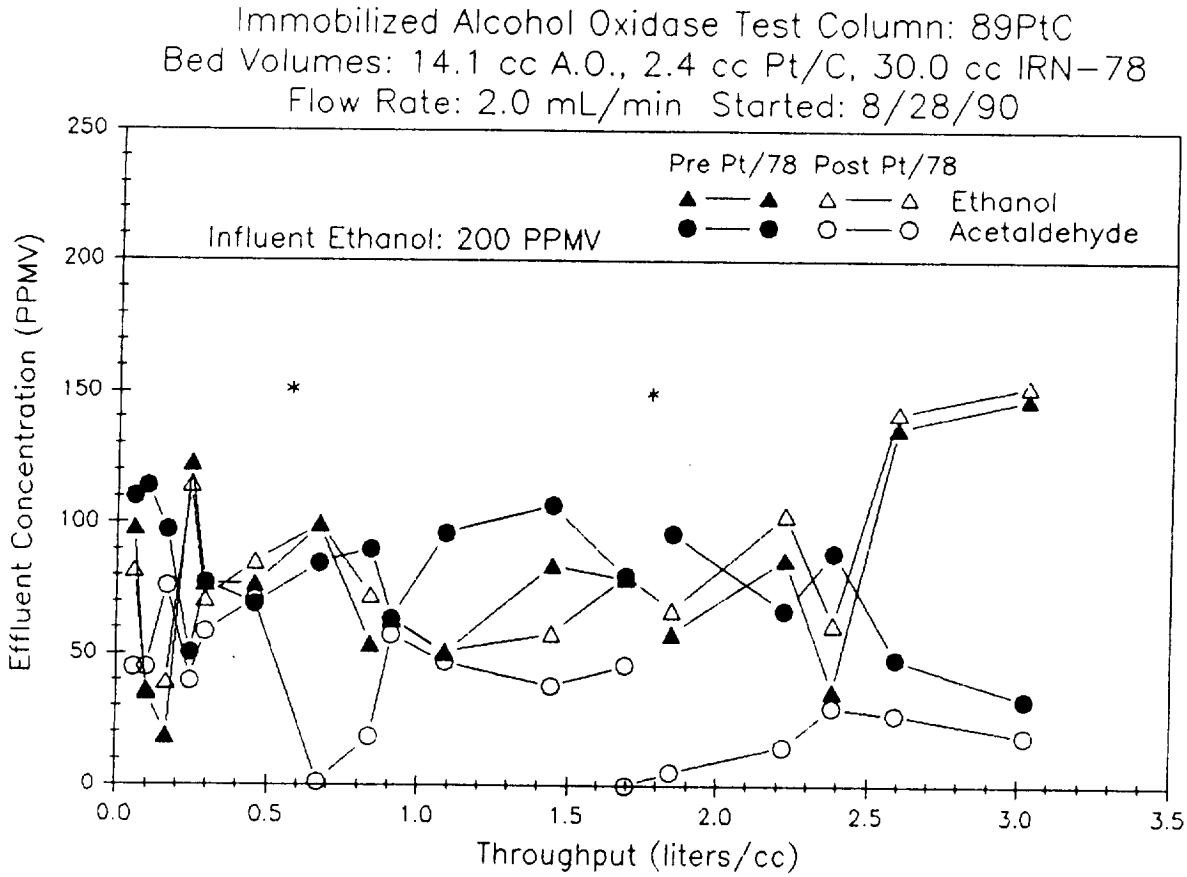

Immobilized Alcohol Oxidase Test Column : 80Pt20
Column Volume: 4.8 cc Flow Rate: 2.5 mL/min
Started: 8/15/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0
1
2
3
4
5
6
7
8
9
10

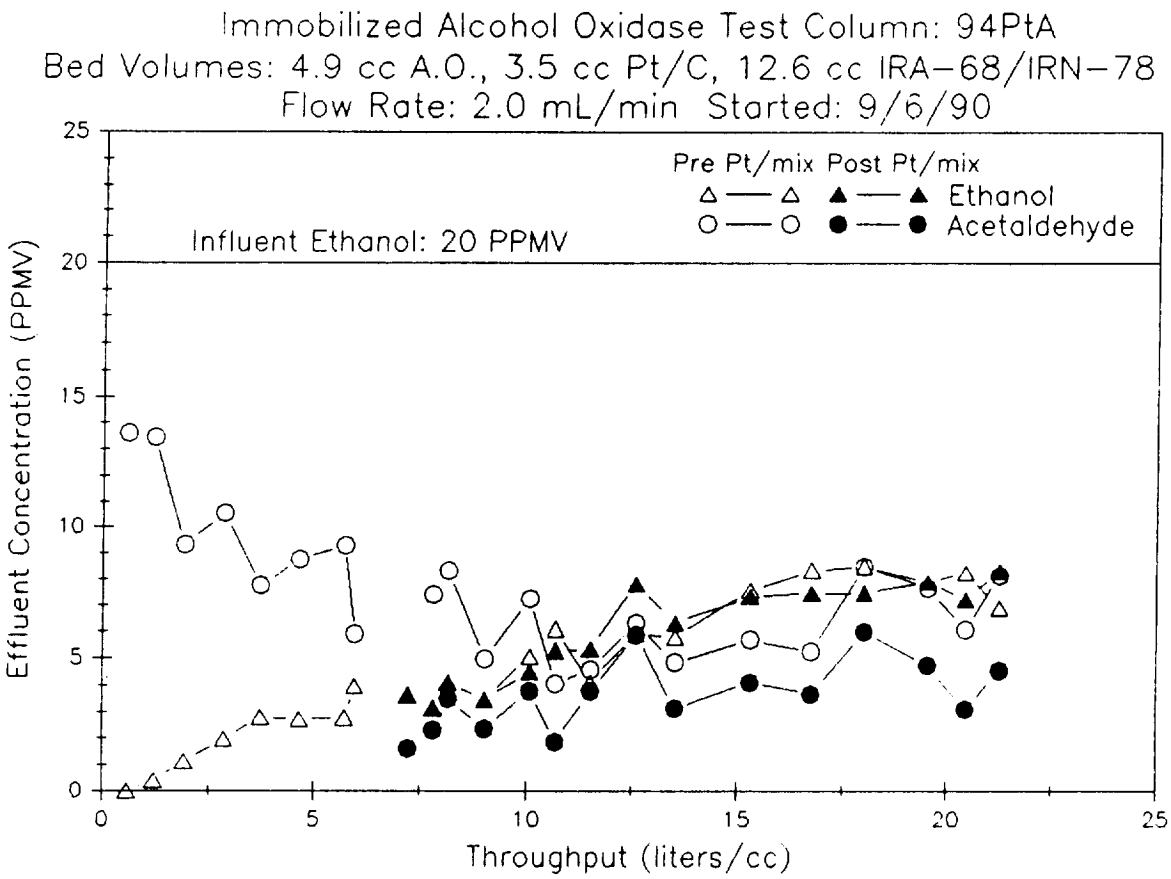

Immobilized Alcohol Oxidase Test Column : 85-A
Column Volume: 5.1 cc Flow Rate: 2.0 mL/min
Started 8/22/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0

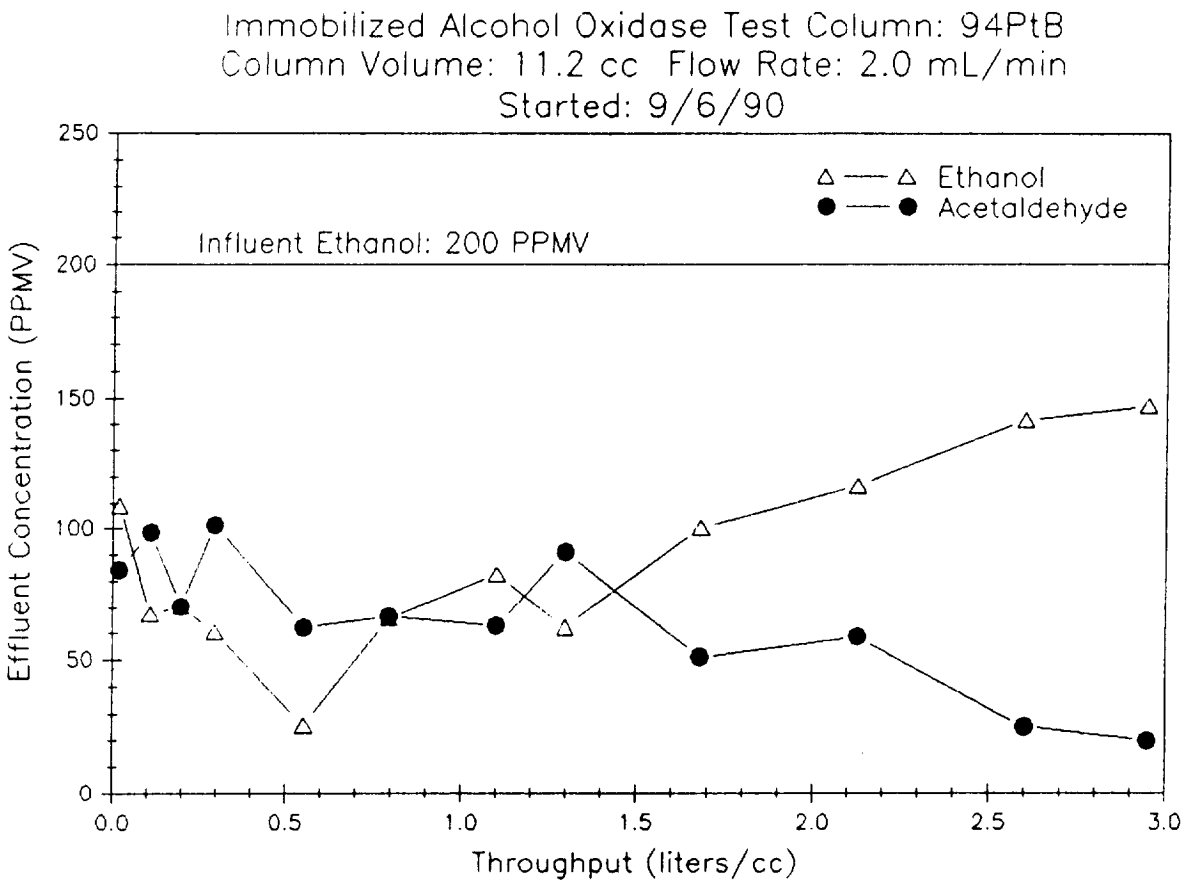

Immobilized Alcohol Oxidase Test Column : 85-B
Column Volume: 5.1 cc Flow Rate: 2.5 mL/min
Started 8/22/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0

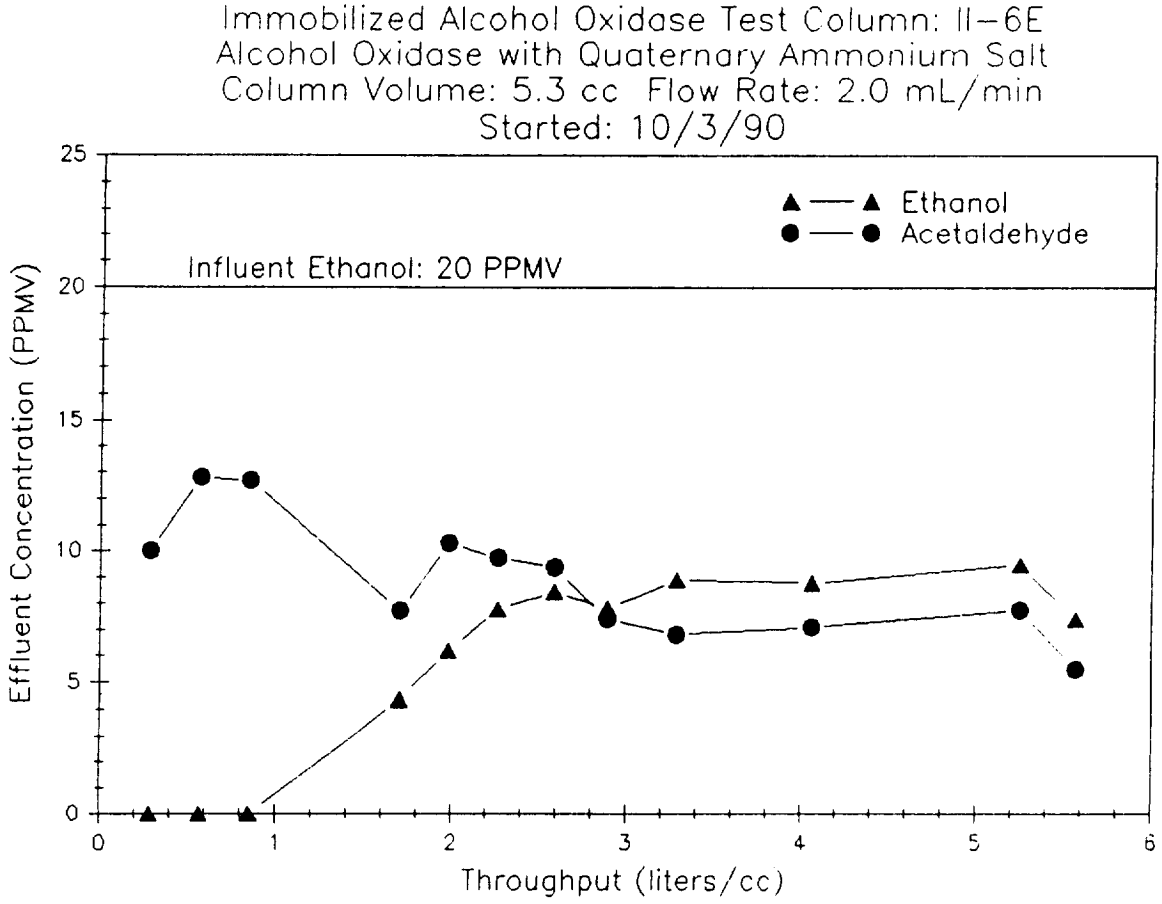

Immobilized Alcohol Oxidase Test Column: 89PtA
Bed Volumes: 5.1 cc A.O., 2.4 cc Pt/C, 5.8 cc IRA-68
Flow Rate: 2.0 mL/min Started: 8/28/90
Pre Pt/68 Post Pt/68
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17

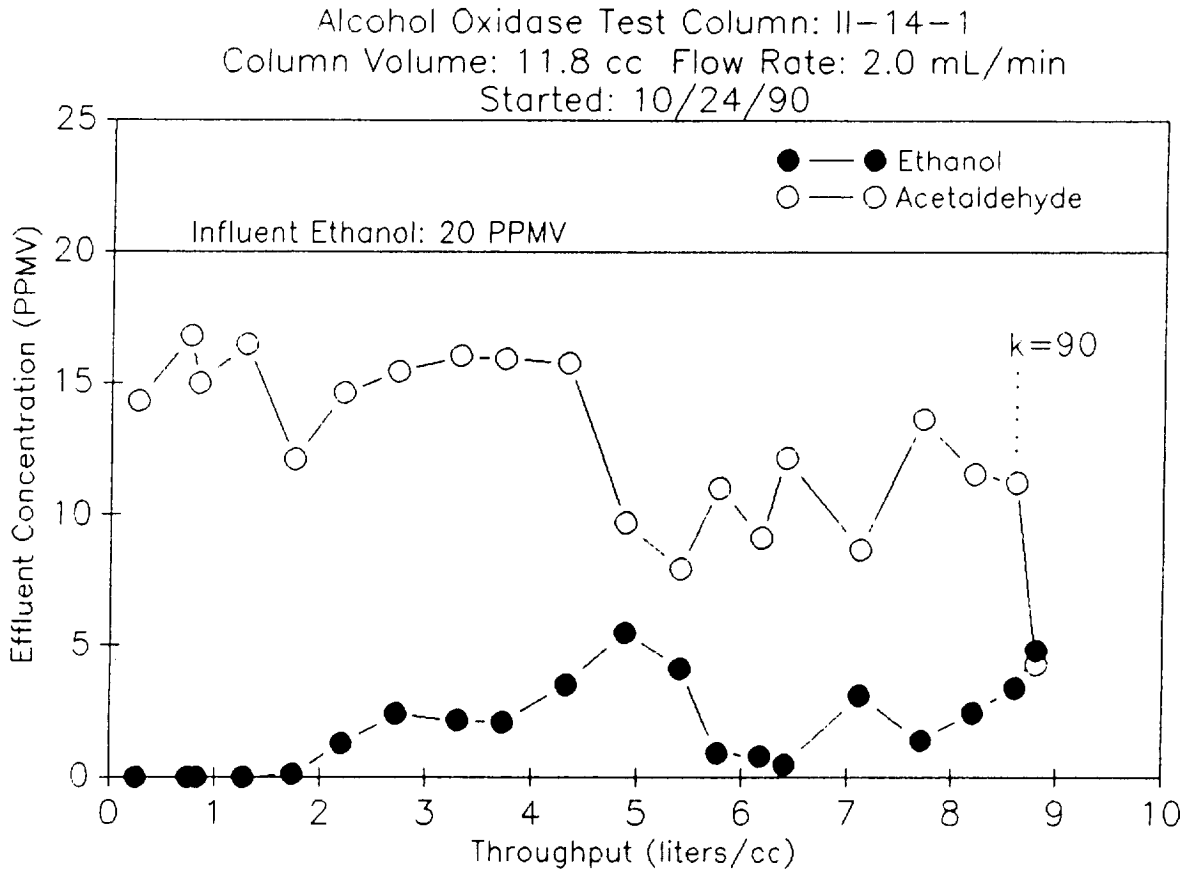

Alcohol Oxidase Test Column: 89PtB
Column Volume: 14.0 CC Started 8/28/90
Flow Rate: 2.0 mL/min
Ethanol
Acetaldehyde
20 ppm ethanol influent
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
30

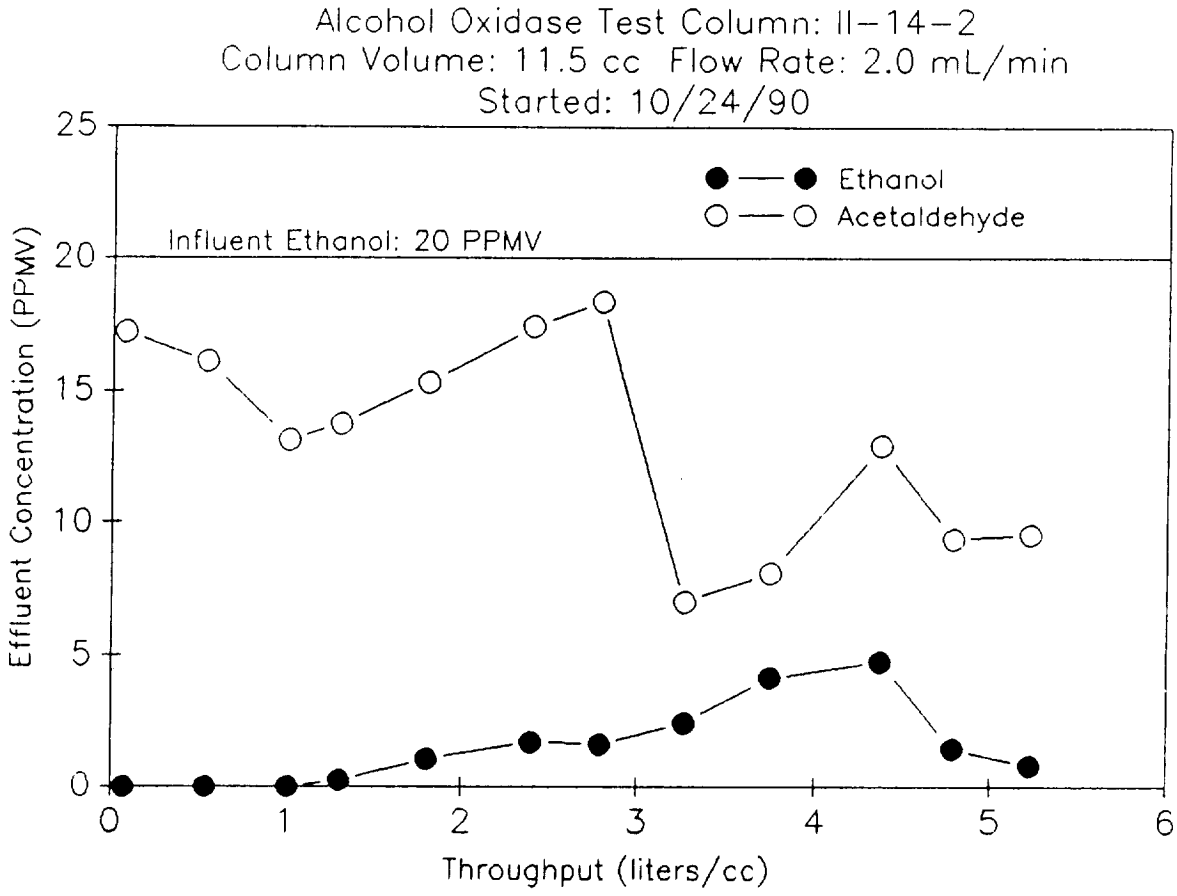

Immobilized Alcohol Oxidase Test Column: 89PtC
Bed Volumes: 14.1 cc A.O., 2.4 cc Pt/C, 30.0 cc IRN-78
Flow Rate: 2.0 mL/min Started: 8/28/90
Pre Pt/78 Post Pt/78
Ethanol
Acetaldehyde
Influent Ethanol: 200 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
250
200
150
100
50
0
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
* IRN-78 Bed Replaced

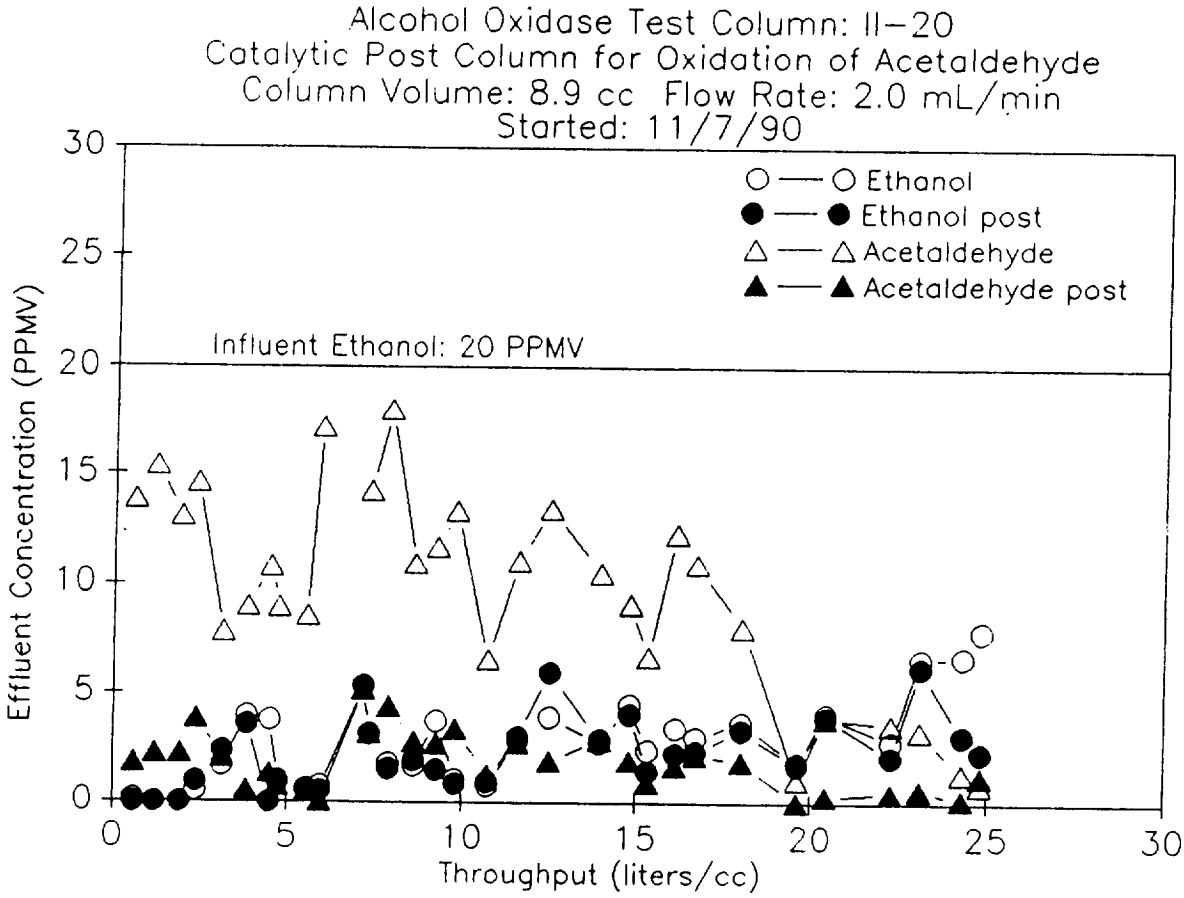

Immobilized Alcohol Oxidase Test Column: 94PtA
Bed Volumes: 4.9 cc A.O., 3.5 cc Pt/C, 12.6 cc IRA-68/IRN-78 mix
Flow Rate: 2.0 mL/min Started: 9/6/90
Pre Pt/mix Post Pt/mix
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25

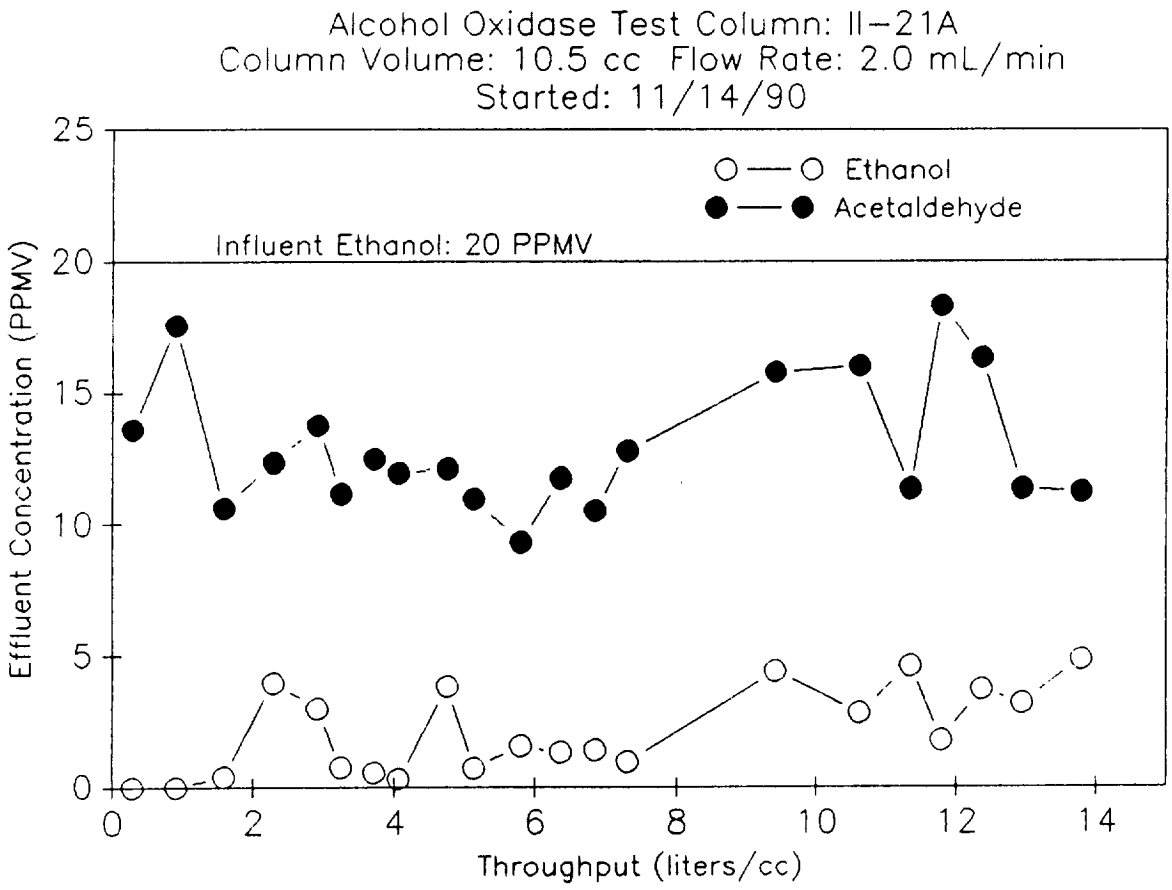

Immobilized Alcohol Oxidase Test Column: 94PtB
Column Volume: 11.2 cc Flow Rate: 2.0 mL/min
Started: 9/6/90
Ethanol
Acetaldehyde
Influent Ethanol: 200 PPMV
Effluent Concentration (PPMV)
250
200
150
100
50
0
0.0
0.5
1.0
1.5
2.0
2.5
3.0
Throughput (liters/cc)

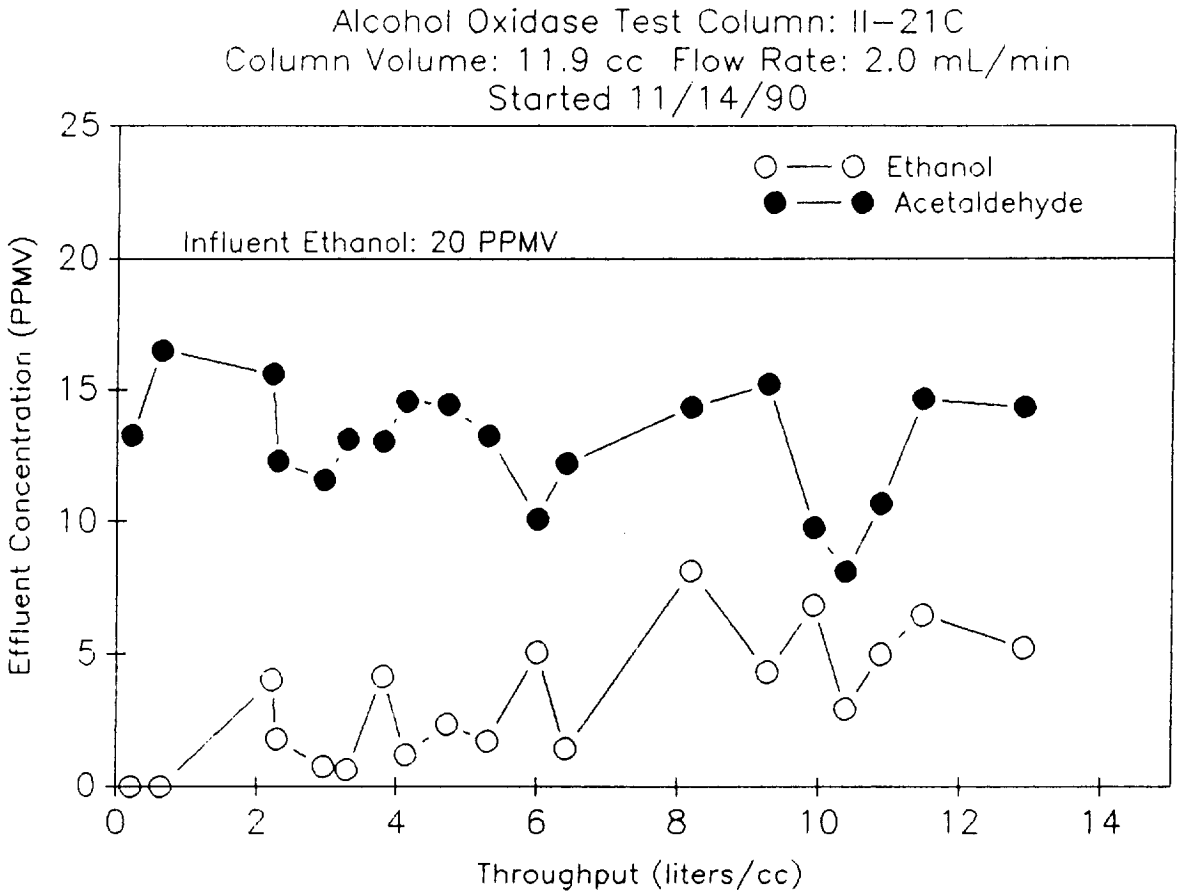

Immobilized Alcohol Oxidase Test Column: II-6E
Alcohol Oxidase with Quaternary Ammonium Salt
Column Volume: 5.3 cc Flow Rate: 2.0 mL/min
Started: 10/3/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0
1
2
3
4
5
6

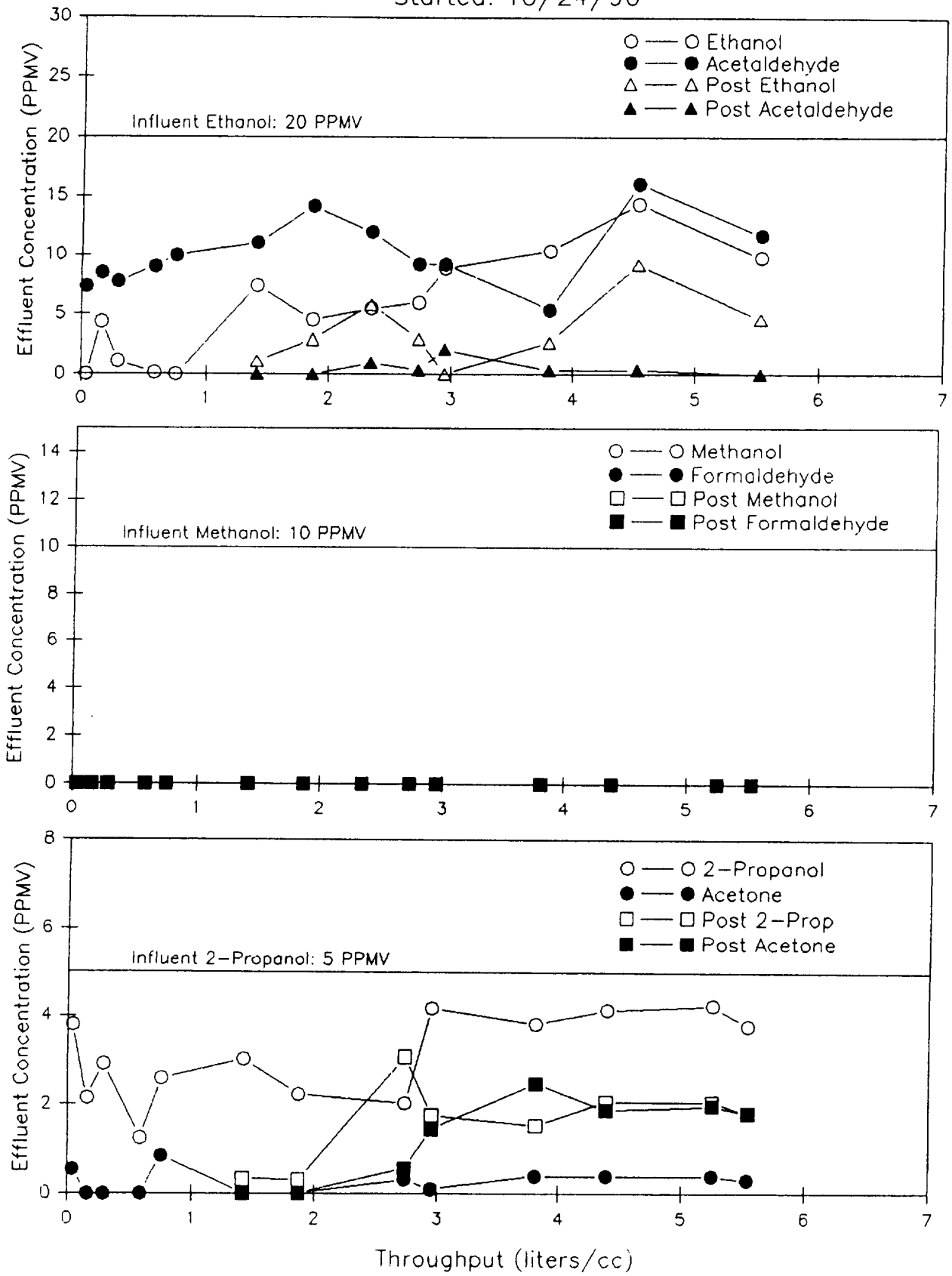

Alcohol Oxidase Test Column: II-14-1
Column Volume: 11.8 cc Flow Rate: 2.0 mL/min
Started: 10/24/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
k=90
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0
1
2
3
4
5
6
7
8
9
10

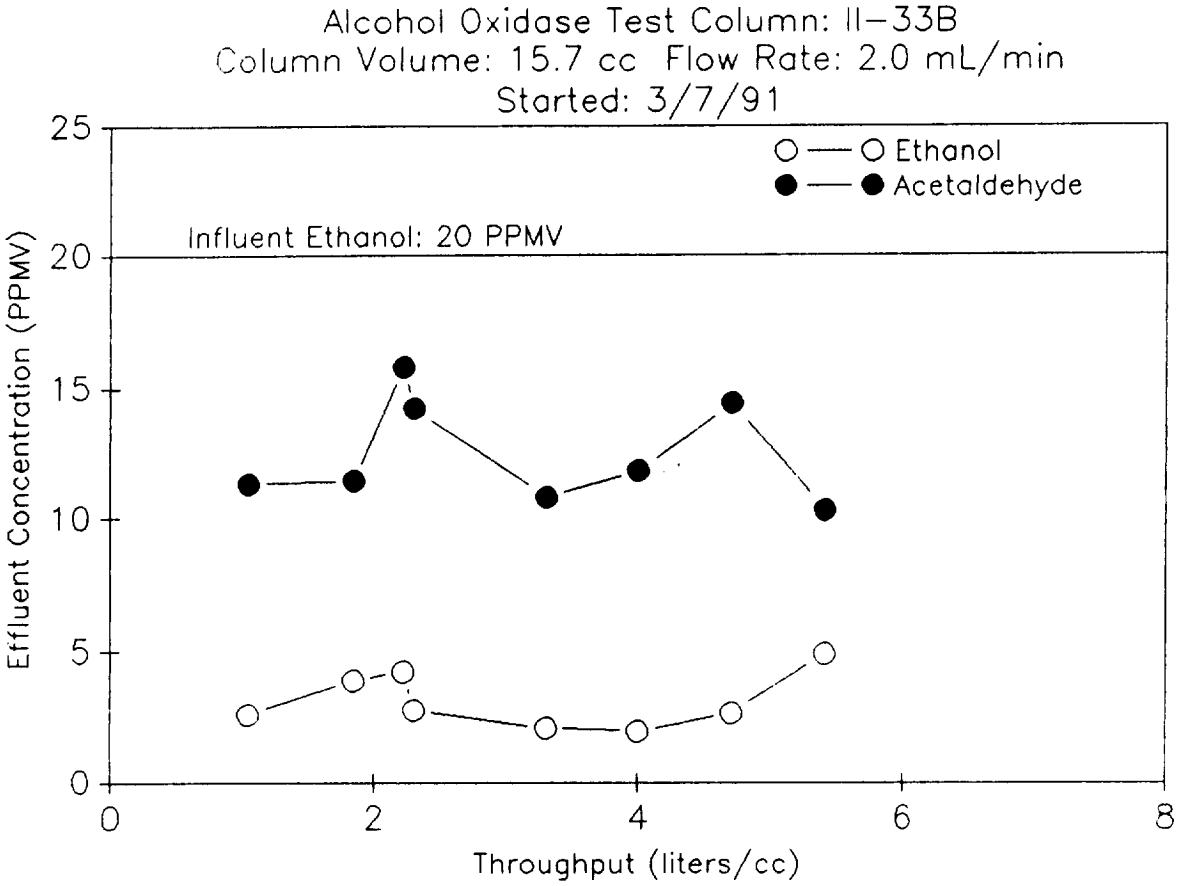

Alcohol Oxidase Test Column: II-14-2
Column Volume: 11.5 cc Flow Rate: 2.0 mL/min
Started: 10/24/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
25
20
15
10
5
0
0
1
2
3
4
5
6
Throughput (liters/cc)

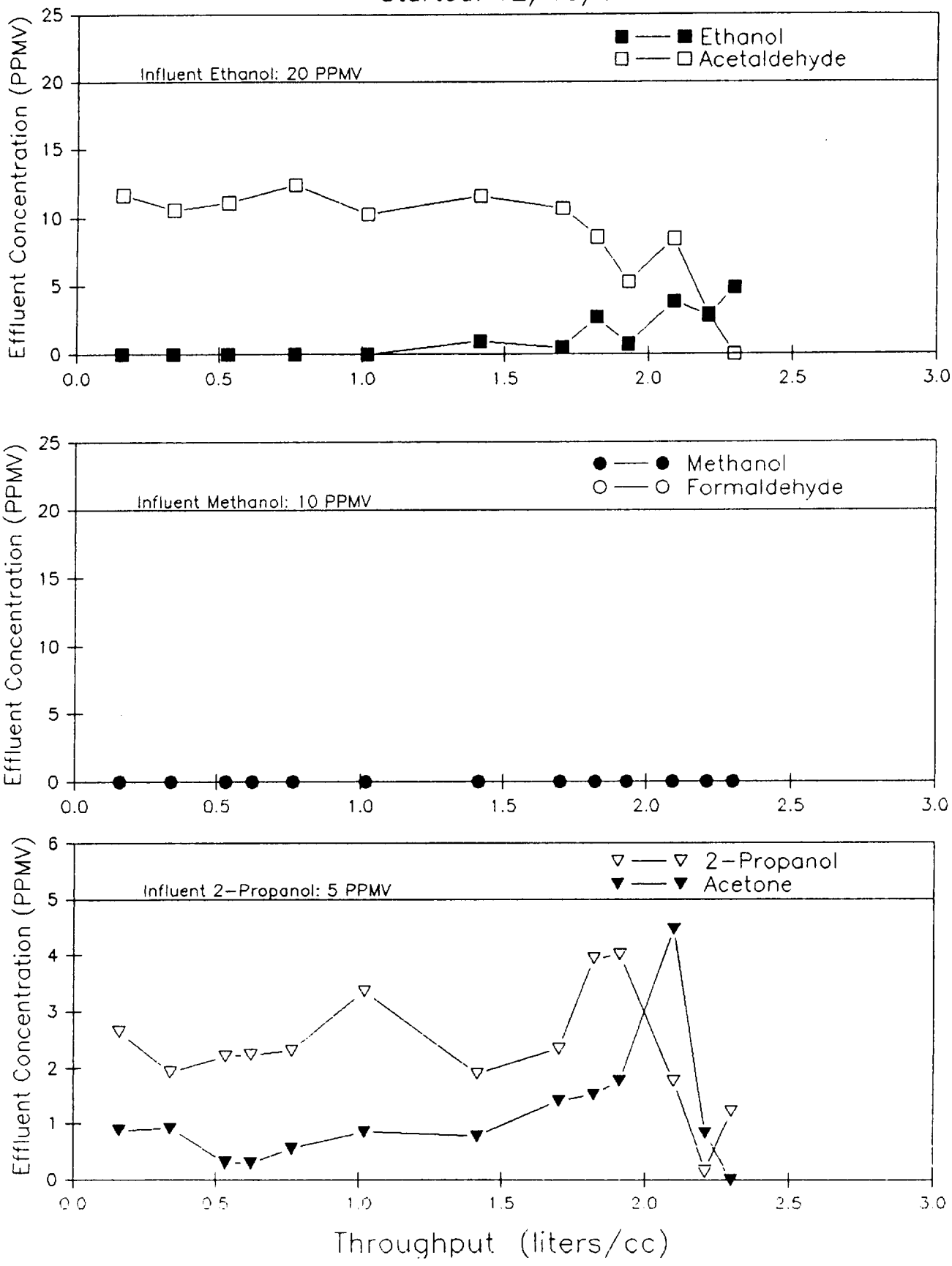

Alcohol Oxidase Test Column: II–20
Catalytic Post Column for Oxidation of Acetaldehyde
Column Volume: 8.9 cc Flow Rate: 2.0 mL/min
Started: 11/7/90
Ethanol
Ethanol post
Acetaldehyde
Acetaldehyde post
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
30

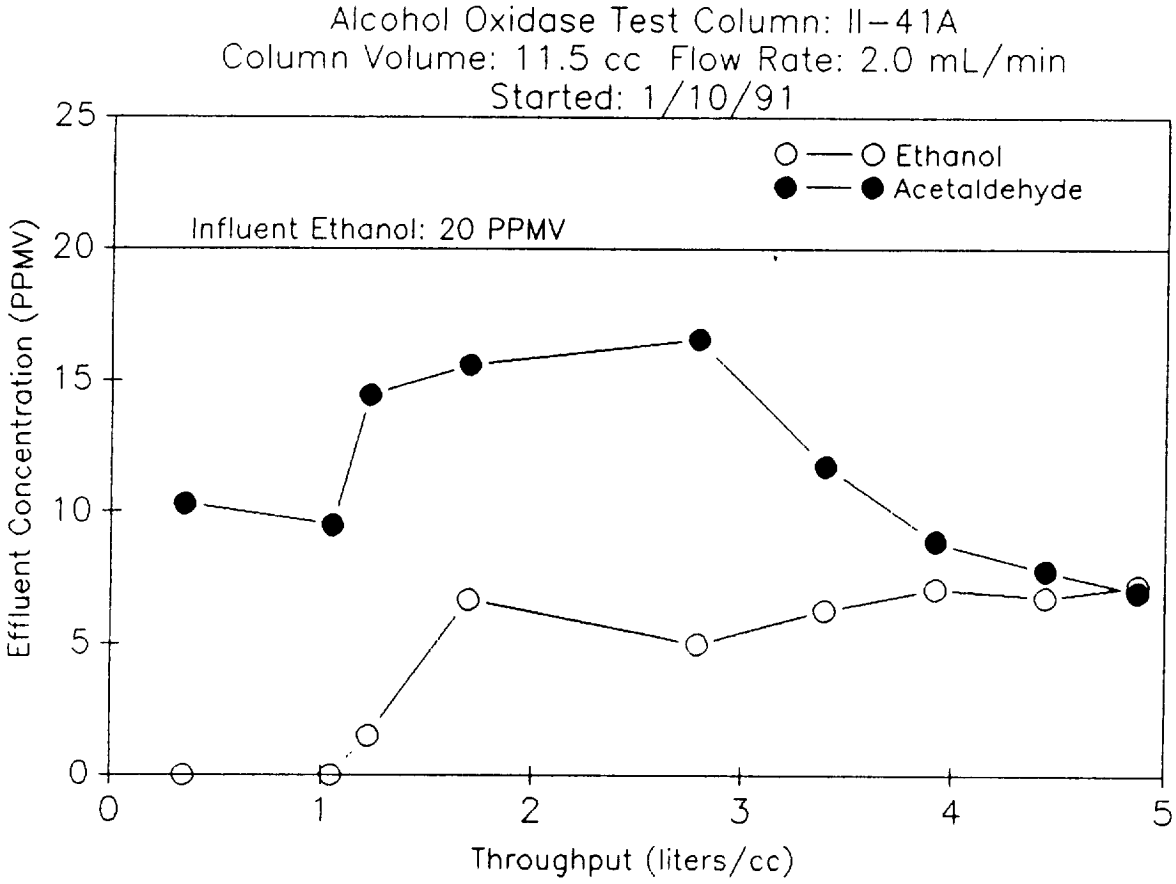

Alcohol Oxidase Test Column: II-21A
Column Volume: 10.5 cc Flow Rate: 2.0 mL/min
Started: 11/14/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0
2
4
6
8
10
12
14

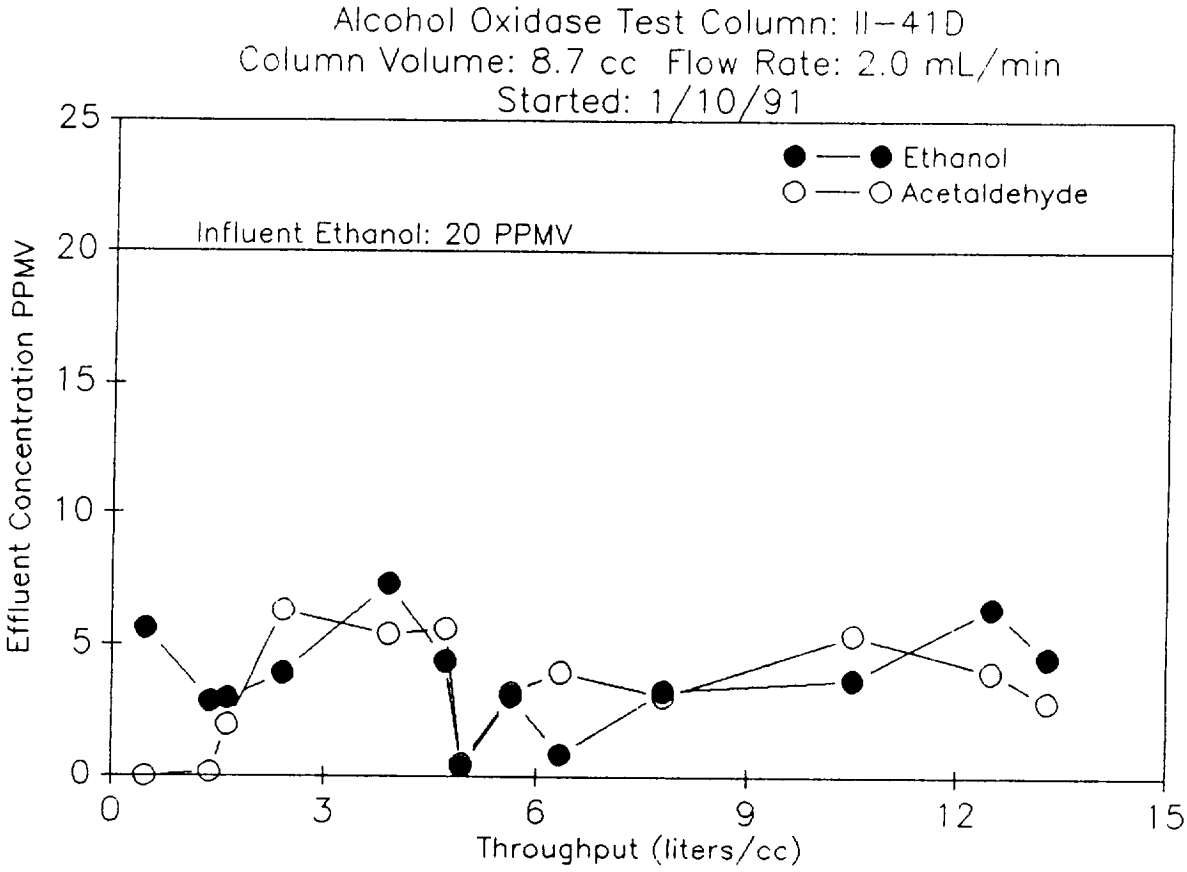

Alcohol Oxidase Test Column: II-21C
Column Volume: 11.9 cc Flow Rate: 2.0 mL/min
Started 11/14/90
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
25
20
15
10
5
0
0
2
4
6
8
10
12
14
Throughput (liters/cc)

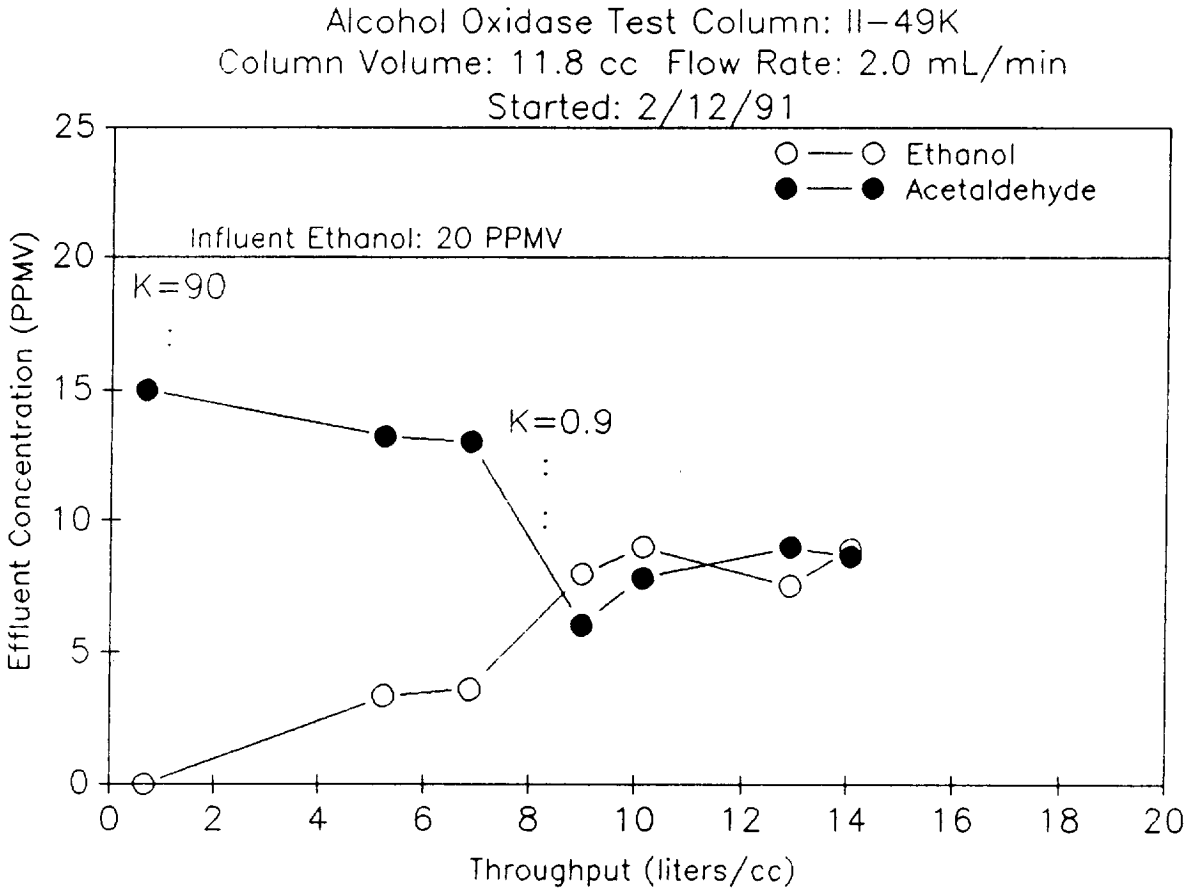

Alcohol Oxidase Test Column: ii-33
Platinum/Carbon Catalyst Post Bed Volume: 31.7 cc
Column Volume: 13.9 cc Flow Rate: 2.0 mL/min
Started: 10/24/90
Effluent Concentration (PPMV)
Influent Ethanol: 20 PPMV
O — O Ethanol
● — ● Acetaldehyde
△ — △ Post Ethanol
▲ — ▲ Post Acetaldehyde
Influent Methanol: 10 PPMV
O — O Methanol
● — ● Formaldehyde
□ — □ Post Methanol
■ — ■ Post Formaldehyde
Influent 2-Propanol: 5 PPMV
O — O 2-Propanol
● — ● Acetone
□ — □ Post 2-Prop
■ — ■ Post Acetone
Throughput (liters/cc)

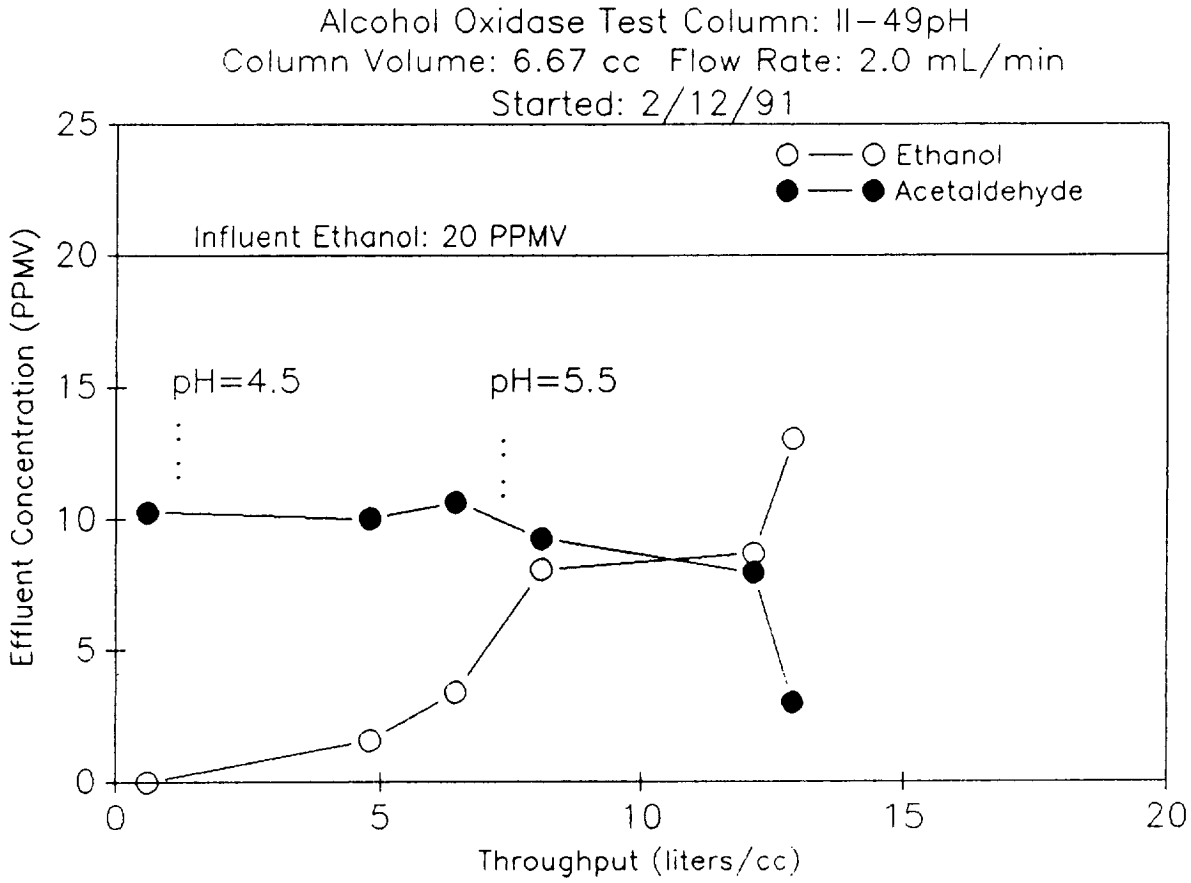

Alcohol Oxidase Test Column: II-33B
Column Volume: 15.7 cc Flow Rate: 2.0 mL/min
Started: 3/7/91
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
25
20
15
10
5
0
0
2
4
6
8
Throughput (liters/cc)

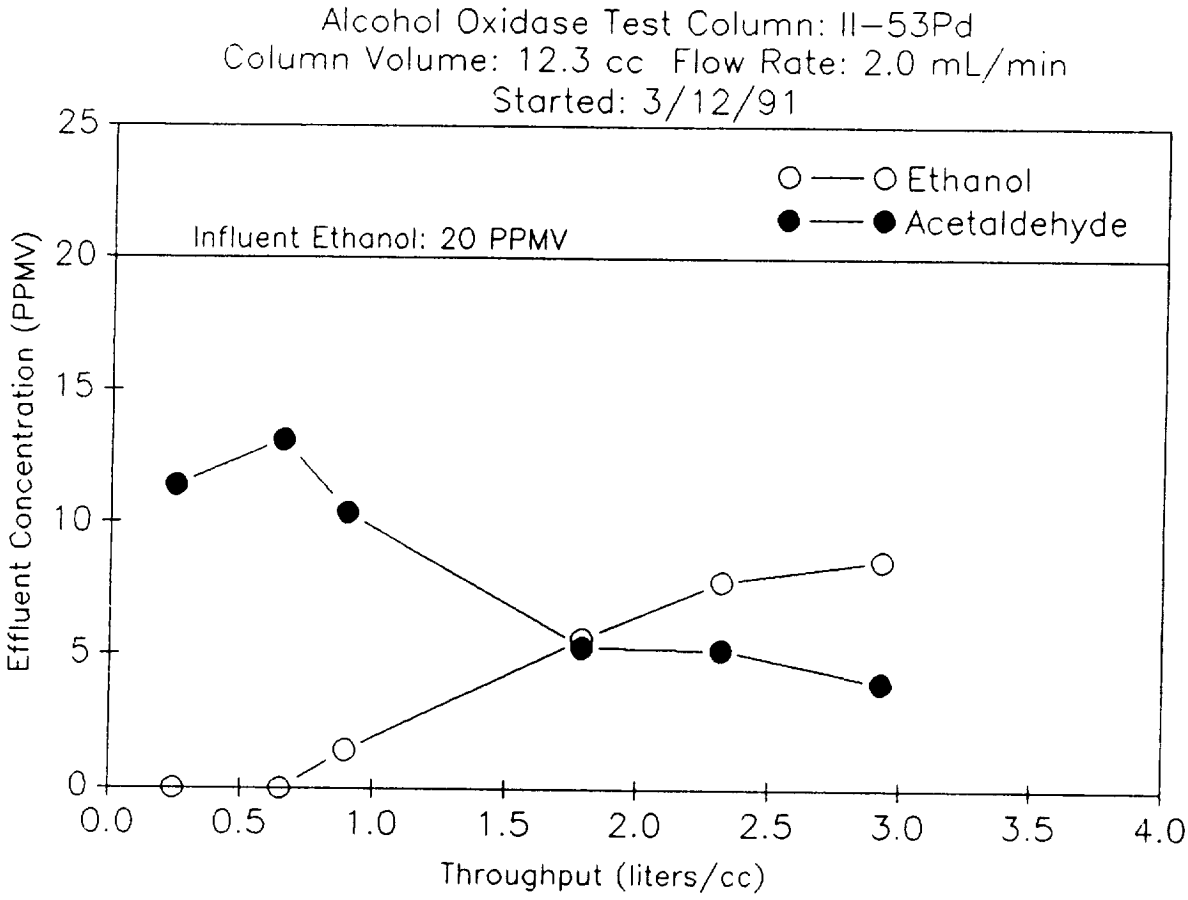

Alcohol Oxidase Test Column: II-35B
Column Volume: 35.3 cc Flow Rate: 2.0 mL/min
Started: 12/15/90
Effluent Concentration (PPMV)
Influent Ethanol: 20 PPMV
Ethanol
Acetaldehyde
Influent Methanol: 10 PPMV
Methanol
Formaldehyde
Influent 2-Propanol: 5 PPMV
2-Propanol
Acetone
Throughput (liters/cc)

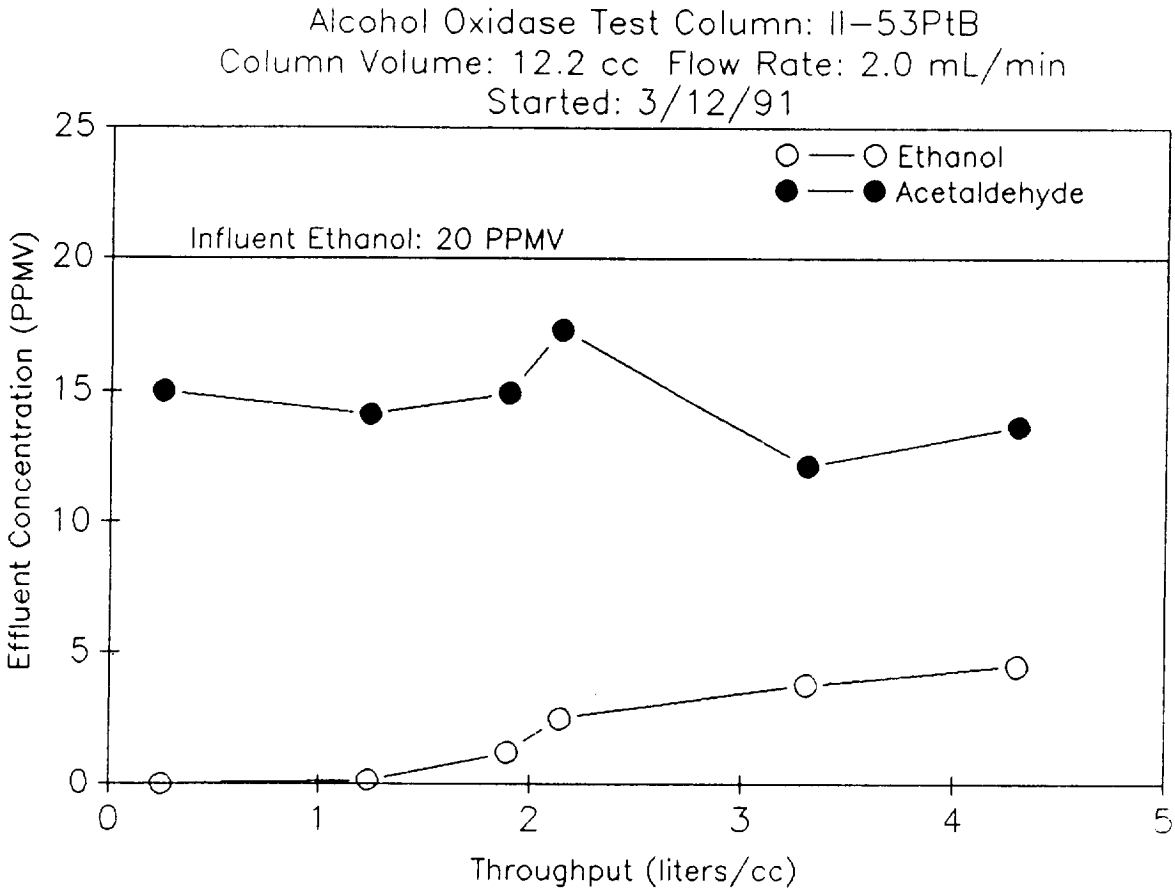

Alcohol Oxidase Test Column: II-41A
Column Volume: 11.5 cc Flow Rate: 2.0 mL/min
Started: 1/10/91
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0
1
2
3
4
5

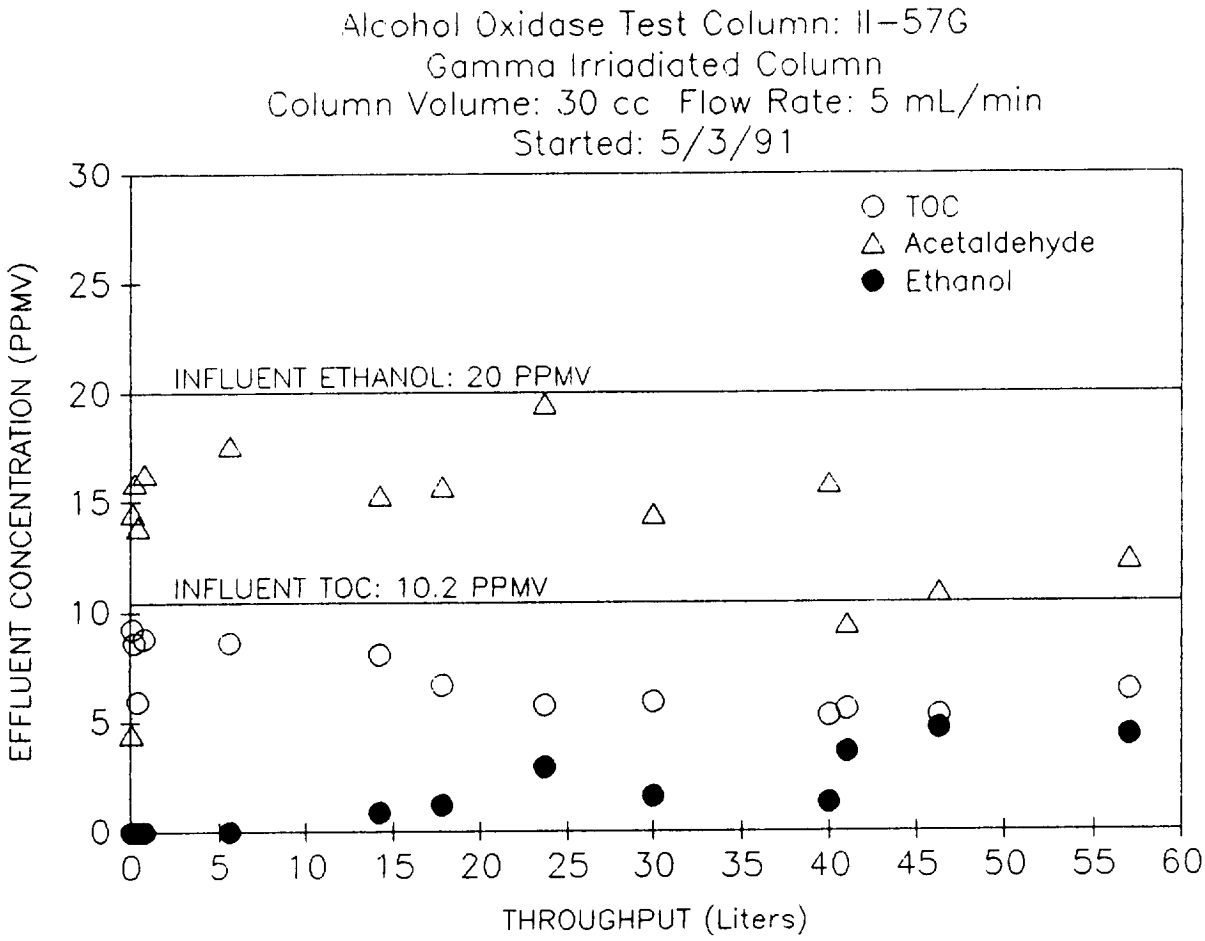

Alcohol Oxidase Test Column: II-41D
Column Volume: 8.7 cc Flow Rate: 2.0 mL/min
Started: 1/10/91
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration PPMV
25
20
15
10
5
0
0
3
6
9
12
15
Throughput (liters/cc)


Alcohol Oxidase Test Column: II-49K
Column Volume: 11.8 cc Flow Rate: 2.0 mL/min
Started: 2/12/91
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
K=90
K=0.9
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25
0
2
4
6
8
10
12
14
16
18
20


Alcohol Oxidase Test Column: II-49pH
Column Volume: 6.67 cc Flow Rate: 2.0 mL/min
Started: 2/12/91
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
pH=4.5
pH=5.5
Effluent Concentration (PPMV)
Throughput (liters/cc)
0
5
10
15
20
25


Alcohol Oxidase Test Column: II–53Pd
Column Volume: 12.3 cc Flow Rate: 2.0 mL/min
Started: 3/12/91
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
25
20
15
10
5
0
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
Throughput (liters/cc)


Alcohol Oxidase Test Column: II–53PtB
Column Volume: 12.2 cc Flow Rate: 2.0 mL/min
Started: 3/12/91
Ethanol
Acetaldehyde
Influent Ethanol: 20 PPMV
Effluent Concentration (PPMV)
25
20
15
10
5
0
0
1
2
3
4
5
Throughput (liters/cc)


Alcohol Oxidase Test Column: II-57G
Gamma Irriadiated Column
Column Volume: 30 cc Flow Rate: 5 mL/min
Started: 5/3/91
TOC
Acetaldehyde
Ethanol
INFLUENT ETHANOL: 20 PPMV
INFLUENT TOC: 10.2 PPMV
EFFLUENT CONCENTRATION (PPMV)
THROUGHPUT (Liters)
0
5
10
15
20
25
30
35
40
45
50
55
60

APPENDIX D

Appendix D. Conditions for Urease Test Columns

The urease enzyme was isolated from jack beans, and is obtained as a lyophilized powder. The enzyme was purchased in one of two forms, a raw form, type IX and a highly purified type C-3. The solid was dissolved in water prior to the deposition on the support, incubation typically was for 100 hours at 4°C.

All column testing used 60 mg/L urea as the challenge solution unless otherwise noted. The flow rate was 2.5 mL/min and the columns were tested periodically for ammonia and urea in the effluent stream. Conditions and results of all of these small column tests are discussed in the paragraphs below.

I. A pair of columns were prepared to investigate the difference in ability of the two forms of urease. The 9.2 cc beds were both operated at 4.5 mL/min flow rate. Neither column was very effective at converting the 60 mg/L urea influent solution to ammonia. Initially the columns removed 67% of the urea , but both columns were non-functional after 5 L/cc.

II. Another type IX column with a 12.9 cc bed was run at 6.0 mL/min flow rate. This column completely removed urea for 2 L/cc, but again was completely dead before 5 L/cc throughput.

III. A urease column was prepared with a 4.6 cc bed volume. The flow rate was 2.5 mL/min. This column, B/N U900130-1, was started 1/30/90 and run for 78 L/cc. The column did not have a

long duration for complete removal of urea, but performed well. There was a steady decline in ability over the 8 months of operation. This decline may be due to a gradual loss of urease enzyme from the support media.

IV. A 5.0 cc bed was prepared and operated at several different flow rates. The column was started at a 3.0 mL/min flow rate and run for 1.6 L/cc. After this the column was operated at 2.5 mL/min for 5.4 L/cc. The extended contact time elicited an improvement in the column performance. Once the column began to fail, the flow rate was decreased to 0.5 mL/min. The column improved once again, but the rapidly fell off during the next 4 L/cc. At this point the column was discontinued.

V. A test column prepared with type IX urease by the dilute titanium method was operated, beginning on 4/3/90. The column was the best performing support from a series of miniature columns, which tested variations in the immobilization process. This media was prepared using 1% hexanediamine as the linkage agent, 1.25% glutaraldehyde as the coupling agent and an enzyme incubation period of 24 hours. The column (13EU) was washed with a phosphate buffer solution prior to elution with 60 mg/L urea.

The column removed all the urea for 2.4 L/cc, then fell off gradually through the next 4.8 L/cc. The column was removing only 40% of the urea when it was halted after 7.2 L/cc.

VI. Another urease column was prepared by the same method as above, except that the type IX urease was dissolved in phosphate buffer rather than water. This column (20H+U) functioned similar

to the one above, converting all the urea to ammonia for 2.5 L/cc, then rapidly losing effectiveness.

VII. A series of three columns (II-36) was prepared by the titanium method using 1% ethylenediamine as the linkage agent. The enzyme incubation period was 65 hours, the deposition taking place in phosphate buffer. The 3.5 cc columns were started on 4/30/91. Each column had a different concentration of urea pumped through at 2.5 mL/min flow rate. As expected, the urease bed produced ammonia for a longer duration with the lower concentration influent.

VIII. A type C-3 column (40 EU C3) was prepared using the titanium/ethylenediamine method. The enzyme incubation period was 20 hours at 4°C. This 3.4 cc column, begun on 5/1/90, was run at 2.5 mL/min for 13 liters throughput. The column completely converted urea to ammonia through the first 5 L/cc, but started to fail rapidly after 9 L/cc. This column was better than the previous columns, and encouraged further minor changes in the immobilization process.

IX. A study was done, beginning 5/23/90, to assess immobilization processes other than the standard titanium process. A 6.2 cc column was prepared by the silanization method. The incubation period was 19 days! The column (48S) was operated at 2.5 mL/min flow for 13.5 L/cc throughput. The performance showed total urea removal through the first 3.5 L/cc, followed by a gradual decrease in the ability to remove urea through the next 10 L/cc. The extra long incubation period did not appear to contribute to accelerated performance in this test.

A 5.7 cc column (49U) was prepared by the titanium method using 67% hydrazine in water as the linkage agent. The method was based on work done by a Chinese group.[1] Incubation was for 96 hours. The results were similar to the silane column above; complete urea removal for 3 L/cc, followed by gradual degradation through the next 10 L/cc. There was no advantage to using hydrazine in place of the diamine.

X. A 5.5 cc test column (50EC3) with C-3 urease immobilized by the titanium/ethylenediamine method with a 23 day incubation period. The flow rate was 2.5 mL/min, with the column being run for 13.4 L/cc. The bed converted all the urea to ammonia for 4 L/cc, then fell off gradually through the next 9 L/cc.

XI. A pair of columns (57U) were set up to test the effect of iodine on the performance of immobilized urease. The 6.8 cc columns were prepared by the titanium/ethylenediamine method with a 138 hour incubation period. A microbial check valve (MCV) putting out 0.7 PPMV iodine was put on line to the influent urea stream on one of the two columns. The test was started on 6/6/90, and showed that the enzyme bed with iodine functioned for only 1 L/cc before rapid deactivation of the enzyme occurred. The control column operated for 13.2 L/cc, removing urea completely through the first 4 L/cc and following the same gradual decomposition as seen previously.

XII. A column (62U) was prepared which had no linkage agents between the titanium deposited support and the enzyme. The column is a baseline for the immobilization process, as there is really no

way for the enzyme to be tethered to the support. The 7.0 cc bed actually removed urea for the first L/cc, but was totally inactive after 4 L/cc.

XIII. A set of three columns (66U) for direct comparison of immobilization methods was prepared. This series was run after miniature column tests had eliminated several methods from consideration. A 6.2 cc bed was prepared by the silanization process with a 96 hour incubation at 4°C. At the same time, a pair of type IX urease columns, 5.7 cc and 6.2 cc respectively, were prepared by the titanium/ethylenediamine method and were also incubated for 96 hours, one at 4°C, the other at 23°C. The three columns were set up on 6/27/90, and run at 2.5 mL/min.

The silane column removed urea for 3 L/cc and then gradually failed over the course of the next 8 L/cc. The titanium columns each worked well, removing all the urea for nearly 20 L/cc, a significant improvement over past performance. The columns both were at 67% removal of urea when halted after 42 L/cc. This large improvement was likely due to the sequence of washes introduced during the intermediate steps of the immobilization process.

XIV. The titanium method tests described above were repeated with another batch of material (80U), to confirm that the improvement was not just an artifact. Two 5.3 cc beds, one cold, the other at room temperature were prepared by the titanium/ethylenediamine process with a 92 hour incubation time. Begun on 8/6/90, these beds were operated at 2.5 mL/min. The previous performance was reproduced through 40 L/cc.

At the same time, four more columns (80C3) were prepared with the type C-3 urease enzyme by the titanium/ethylenediamine method. These were also incubated at 4°C for 92 hours. Each column contained roughly 5.5 cc media. Two columns were stored at 4°C, the other two were set up on 8/6/90, with 60 mg/L urea and 200 mg/L urea influent respectively. Again, the flow rate was kept at 2.5 mL/min. The 60 mg/L column removed all the urea for 25 L/cc and was over 90% removal for the next 15 L/cc, before a sudden total deactivation. The 200 mg/L column converted all the urea to ammonia for 15 L/cc, then 90% for the next 15 L/cc.

XV. A series of urease columns were prepared with several transition metals deposited onto the support prior to the titanization process. Beds deposited with silver, platinum, copper and tin were tested for both urease ability and biostasic ability. The 6.0 cc columns were eluted with 60 mg/L urea at 2.5 mL/min flow rate. Microbial tests were plated on R2A agar and incubated in a 35°C oven. These tests were started on 8/14/90.

The silver column was the only column that showed no microbes in the effluent stream. It also showed no urease activity. The other metals did not effect the urease performance through 2 L/cc, but showed no reduction in the bacteria counts. The copper and tin columns had lower microbial counts than the platinum and control columns.

XVI. A 6.1 cc bed (87U) was prepared with type IX urease by the titanium method with a 92 hour incubation period. The column, designed to test a new batch of support, was run at 2.0 mL/min for

20 L/cc. The bed, begun on 8/20/90, converted nearly all the urea to ammonia for the duration of the test. Another batch test column (93U) was begun on 8/28/91 and run for 20 L/cc at 2.5 mL/min. This different batch of media prepared by the same method also converted over 99% of the urea to ammonia.

XVII. On 8/28/90, one of the 80C3 columns that was stored at 4°C was removed from storage and flushed with a dilute hydrogen peroxide solution (0.03%). The peroxide wash extinguished the enzyme ability to transform urea to ammonia. The change was irreversible, no ammonia was found after 2 liters of flow at 2.5 mL/min.

XVIII. The fourth 80C3 urease column was tested with 16 mg/L formamide as the influent solution. This column began operation on 9/17/90, with the results monitored by HPLC. The column converted only 20 % of the formamide to ammonia and CO_2 through 800 mL total throughput. The ammonia-nitrogen tests showed much more ammonia than could be accounted for, implying that formamide is an interferent toward ammonia testing.

XIX. A pair of columns (II-17) started 10/20/90 were prepared with type IX urease by the titanium/ethylenediamine method with a 164 hour incubation period. The first column, a 7.4 cc bed challenged with 60 mg/L urea at 2.5 mL/min, showed excellent activity through 22 L/cc, then gradually fell off through the next 9 L/cc. The column was still removing over 80% of the urea when the test was stopped after 31 L/cc throughput.

The second column from this batch was tested with 500 mg/L

urea as the influent. This 5.9 cc bed, started 10/30/90 was operated at 2.1 mL/min flow rate for 20 L/cc. The column converted all the urea to ammonia through the first 10 L/cc, then gradually fell off during the next 10 L/cc. The final sample monitored showed a decrease to 50% efficiency.

XX. Another column was set up on 11/27/90 to test another new batch of support media. The bed was prepared with 2% ethylene-diamine on titanium, then 5% glutaraldehyde and type IX urease. It was incubated for 117 hours. The 5.5 cc column (II-21) was operated with 60 mg/L at 2.5 mL/min. After 6 months of testing, this column has completely removed the urea for 47 L/cc and is still in operation. This approach toward immobilizing urease produces consistent performance with each batch of support media.

XXI. A pair of urease columns (II-33) were prepared with type IX urease by the titanium method. The columns were started on 12/17/90 after a 143 hour incubation. After 20 liters of flow at 2.5 L/cc, the 60 mg/L influent streams were altered to test the effect of pH and conductivity variation on the urease performance. The pH column was incrementally lowered in pH by one unit after about 5.5 L/cc flow. The column has completely converted urea to ammonia after each change, down to a pH = 2.5. The conductivity column was continually increased in K by the addition of NaCl at roughly the same intervals, up to K = 580 millimho. Both columns currently have had 23 L/cc total throughput with no loss of activity for the urease enzyme.

XXII. A 5.5 cc urease bed was set up with an MCV-RT bed

following it in the same tube. The 60 mg/L urea influent solution was flowed through both beds at 2.0 mL/min, then tested for urea and ammonia. The column, started 12/24/90, ran for 19 L/cc and was 90% effective when flow was stopped. There was a small amount of deactivation of the enzyme caused by back flow of iodine, but the total result is encouraging.

XXIII. A set of four columns (II-39) was prepared with type C-3 urease on titanium/ethylenediamine support. The incubation period was 115 hours. Each column was designed to address a different question. A batch test on the media, started 1/7/91 was run at 2.2 mL/min with 60 mg/L urea. A cleaner column containing a 100 cc cation exchange bed in H^+ form, 25 cc anion exchange bed in OH^- form and 15 cc MCV-RT resin was added as a means for removing the ammonia generated by the enzyme bed. The column converted urea to ammonia for 27 L/cc without failure, while the cleaner column removed ammonia for the first 13 L/cc of this test. After 18 L/cc, 20 ppmv ethanol was added to the influent stream, with no effect on the column performance.

XXIV. Two columns from this batch were tested with much higher concentrations of urea. The first 5.6 cc bed was challenged with 1000 mg/L urea at 2.5 mL/min flow rate from 1/7/91 to the present. It has consistently removed over 95% for a duration of 30 L/cc ! A follow-up 5.5 cc column was started on 3/4/91, with 10,000 mg/L urea flowed at 2.7 mL/min. It showed complete removal for 4 L/cc and 90% removal for 10 L/cc before being rapidly overwhelmed by the 1% concentration.

The last column of this set was stored at 4°C for 2.5 months and then run with 60 mg/L urea at 2.5 mL/min. Through 11.5 L/cc, the 6.9 cc column converted all the urea to ammonia.

XXV. A 4.5 cc type IX urease column (II-43) was prepared from the same batch of urease enzyme that was packed into the urease unibed delivered to NASA-MSFC in January 1991. The total time period for enzyme incubation was 144 hours. The column was challenged with 60 mg/L urea at 2.5 mL/min for 24 L/cc throughput. This enzyme bed had complete convertion of urea to ammonia for the entire test duration.

XXVI. A large amount of derivatized support was formulated for the next generation of deliverable enzyme beds. A portion of this material was deposited with urease to ensure that the media would meet specifications. The standard titanium method was employed with 3.5% ethylenediamine and 5% glutaraldehyde solutions as the linkage and binding agents. The incubation period with type IX urease was 96 hours. Four 5.5 cc columns (II-53) were prepared, one of which was immediately set up on 3/1/91 and tested with 60 mg/L urea at 2.5 mL/min flow rate. Through 15 L/cc, this column has removed all the urea.

The other three columns were stored at 4°C until used in a test to determine the effect of elevated temperature on the urease system. The initial test, started 4/1/91, required that two 5.6 cc columns be eluted at 2.0 mL/min with 1000 mg/L urea. The control column was held at ambient temperature (23°C) while the test column was incremented in 10°C intervals within a water bath.

The flow was off during temperature changes and reinstated once the column achieved the set temperature. The collected volume between each change was 200 mL.

The elevated temperature column allowed small amounts of urea through at low temperature (40°C - 50°C) and more as the temperature increased. At 70°C, the column started showing much higher levels of urea. The control column showed no urea in the effluent stream. A lifetest of the immobilized urease was conducted at 60°C.

For this lifetest, the control column was split into two 2.9 cc test beds and repacked into 7 mm o.d. glass columns. The flow rates were about 1.0 mL/min. With 1000 mg/L urea influent, the test determined that urease at elevated temperature had a much shorter lifetime. After a total flow volume of 3 liters per column, the 60°C column was allowing 75% of the urea to pass unchanged. The control functioned normally, with less than 1% of the urea in the effluent.

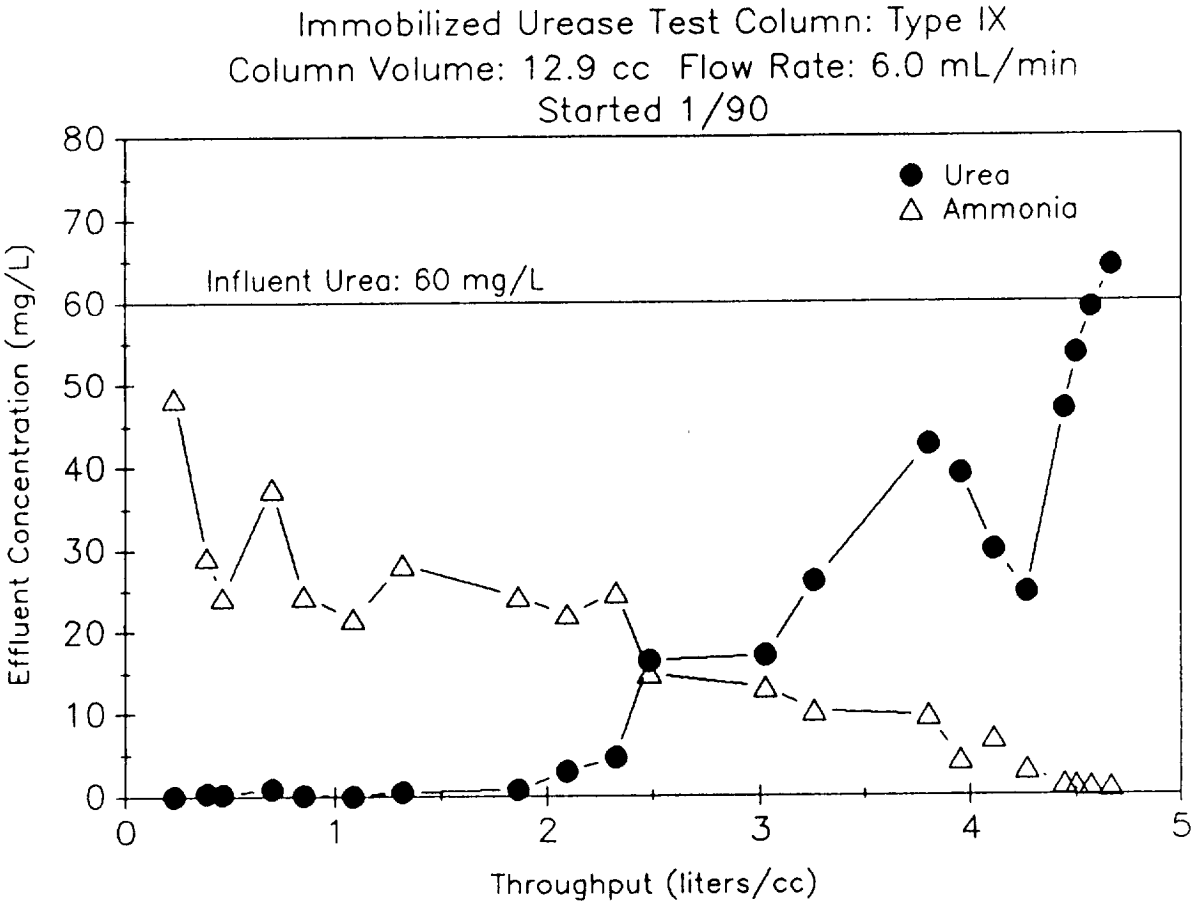
A second elevated temperature test was run at 45°C, with the same conditions as the 65°C test (and the same control column). Both 3 cc columns tested removed all the urea for the first 4 L/cc. This test generated a total throughput of 18 liters per column, at which time the higher temperature column was passing over 40% of the urea in the challenge solution.

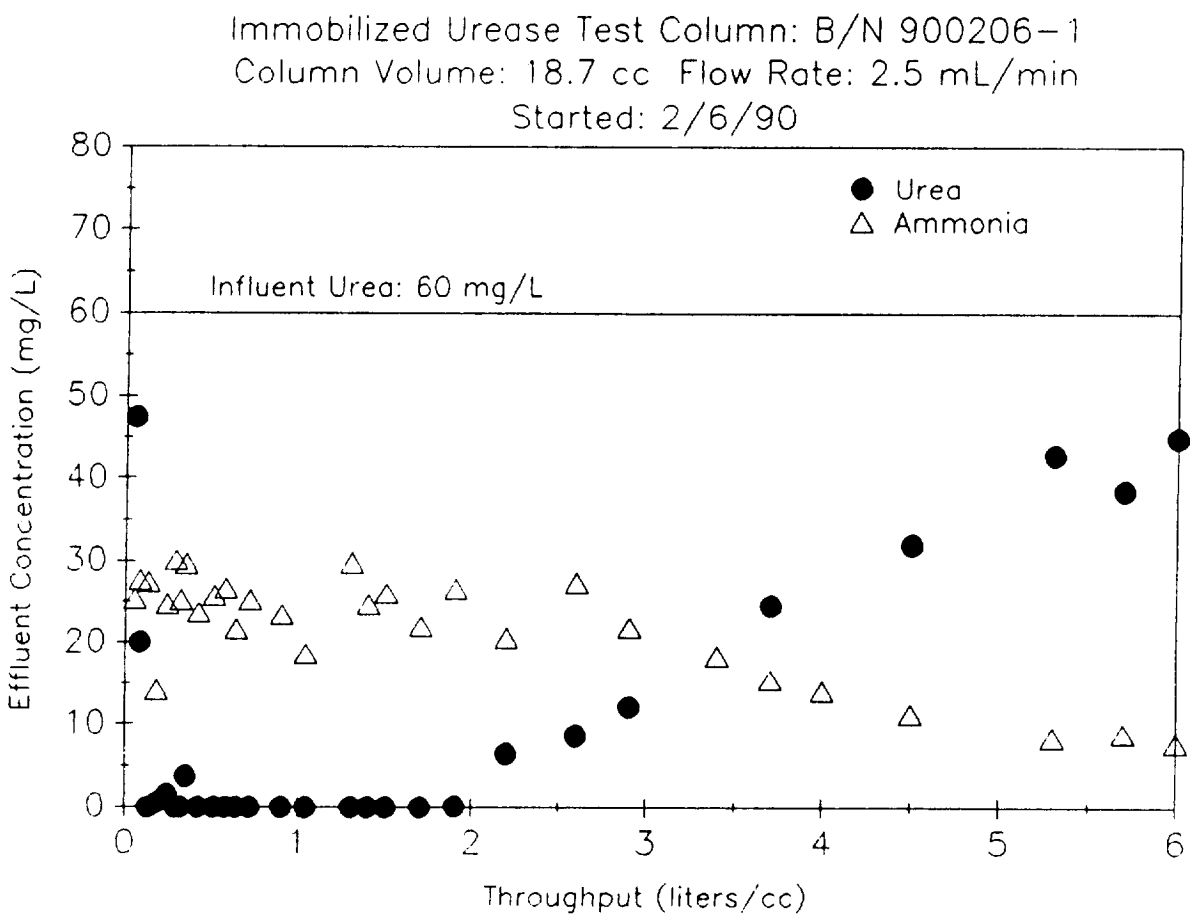
XXVII. Another batch test was prepared on 4/19/91, with material from two different derivitization prcedures. One process involved depositing titanium onto a kilogram of support material at one time. The 6.3 cc columns were prepared using type IX urease

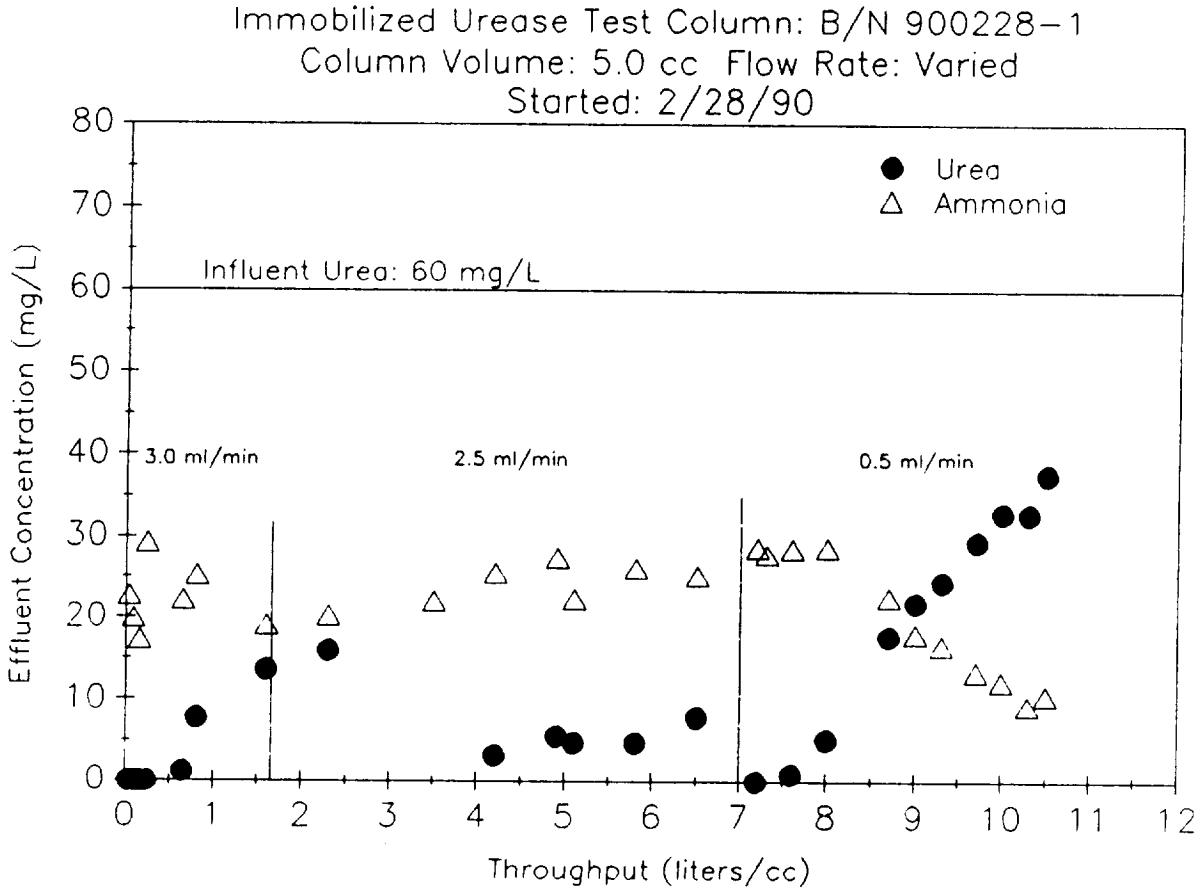
incubated for 96 hours. Initially, both columns removed all of the 60 mg/l urea influent which was flowed at 2.5 mL/min. After 12 liters, the challenge solution was increased to 1000 mg/L urea, to more efficiently test the columns over a shorter period of time. The bulk prep has started to allow some (3.5%) of the total urea feed through after 23 liter volume, whereas the standard prep method removes all the urea.

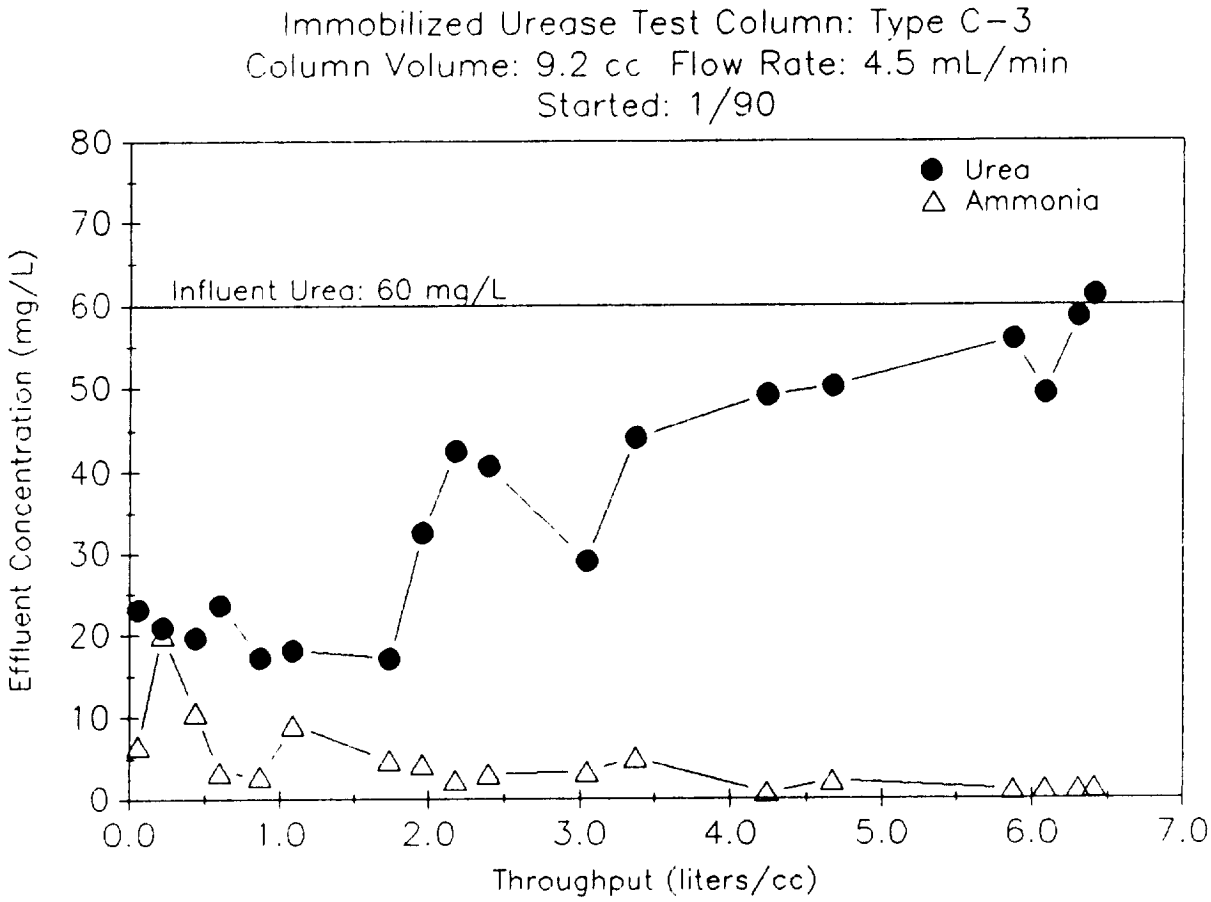
XXVII. A 30 cc column was prepared with type IX urease using a 120 hour incubation period. The column was packed into a 1 inch diameter polycarbonate column and irradiated with 2.5 mrad of gamma radiation. This test is to determine whether radiation used to sterilize the column from microbial contamination affects the performance of the urease enzyme. The column was put onto operation on 5/3/91 with a 60 mg/L feed solution flowed at 4.0 mL/min. Through the first 50 liters of testing, the column has removed all the urea from the feed source, demonstrating that the radiation does not decrease the urease activity. The test will continue to determine the effect on the column lifetime.

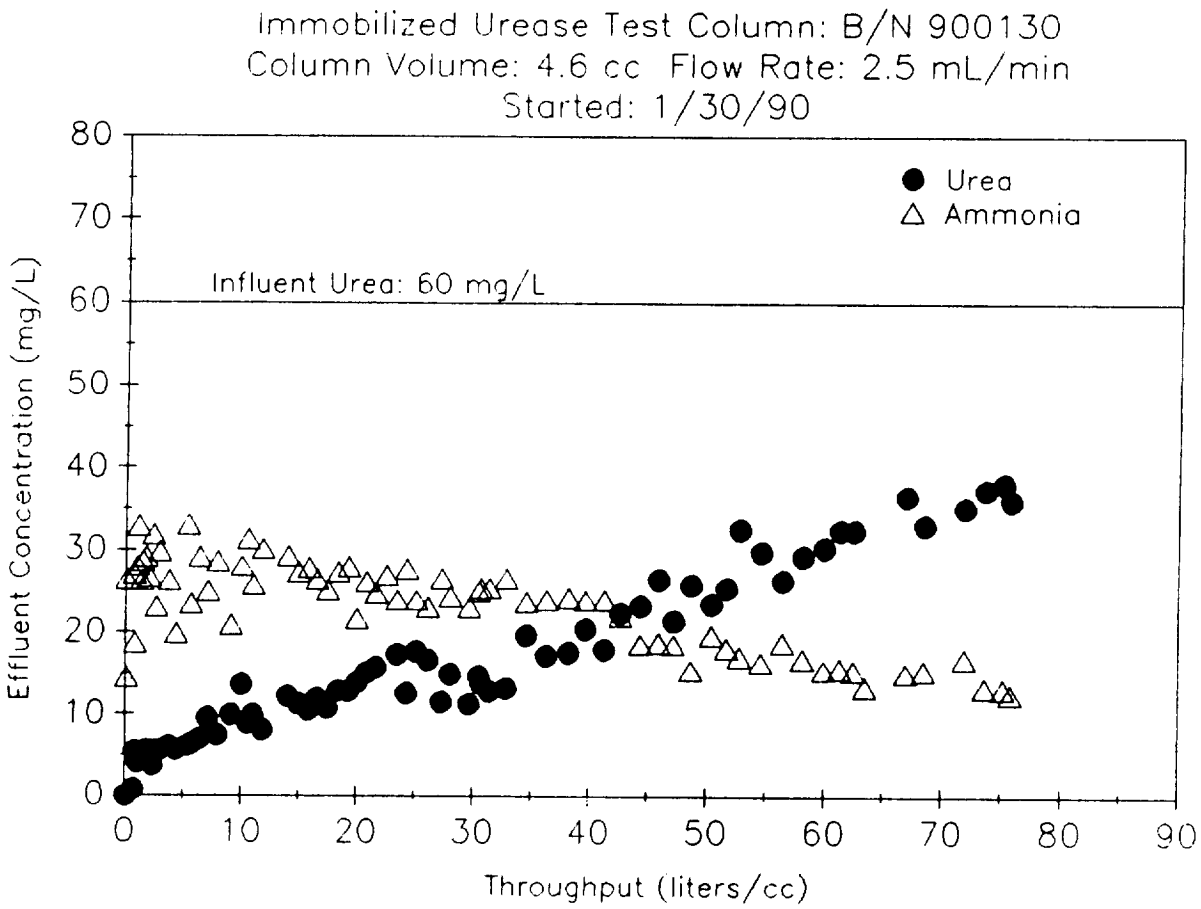
1. Chang-Zhi, C. and Yao-Ting, Y.; Biomaterials, Artificial Cells and Artificial Organs.(1989) 17(3), 329

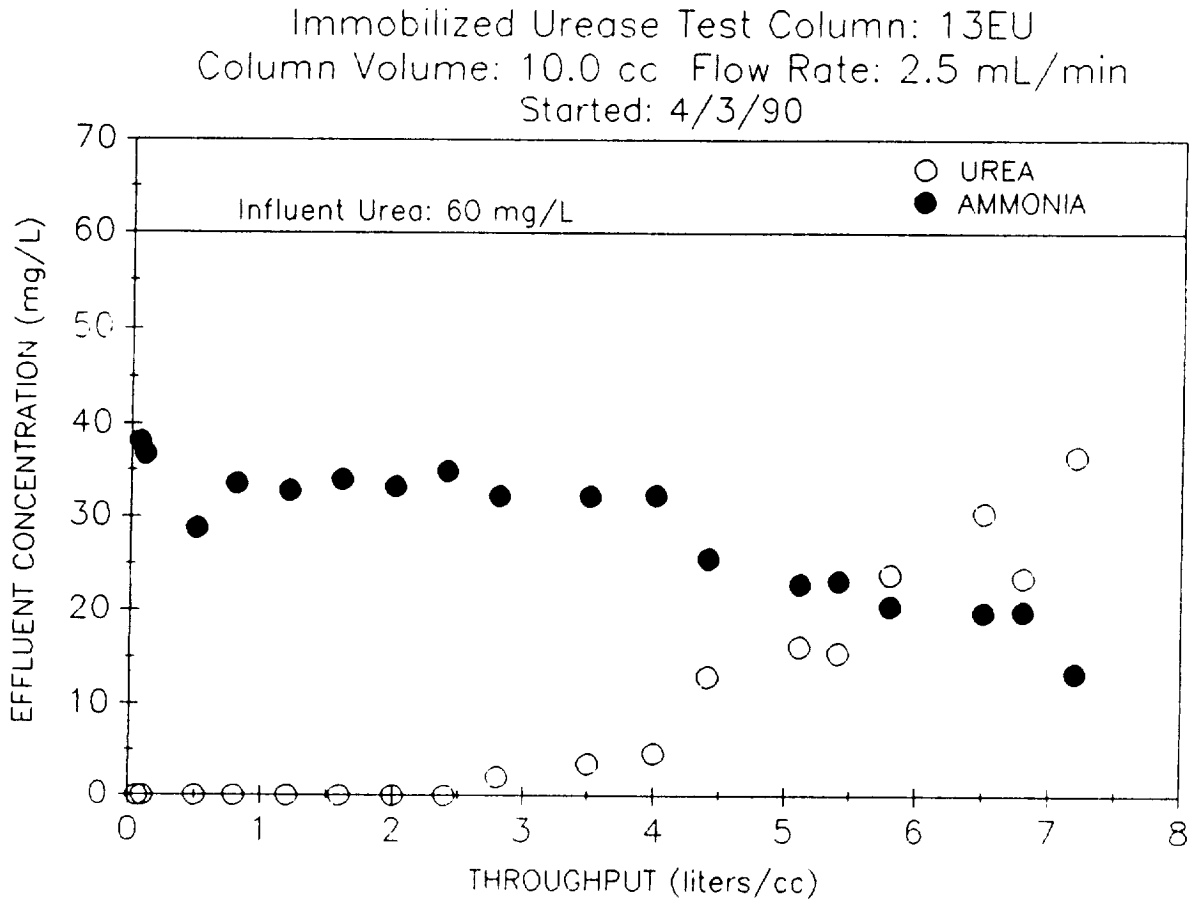

Immobilized Urease Test Column: Type IX
Column Volume: 12.9 cc Flow Rate: 6.0 mL/min
Started 1/90
Urea
Ammonia
Influent Urea: 60 mg/L
Effluent Concentration (mg/L)
Throughput (liters/cc)
0
10
20
30
40
50
60
70
80
0
1
2
3
4
5

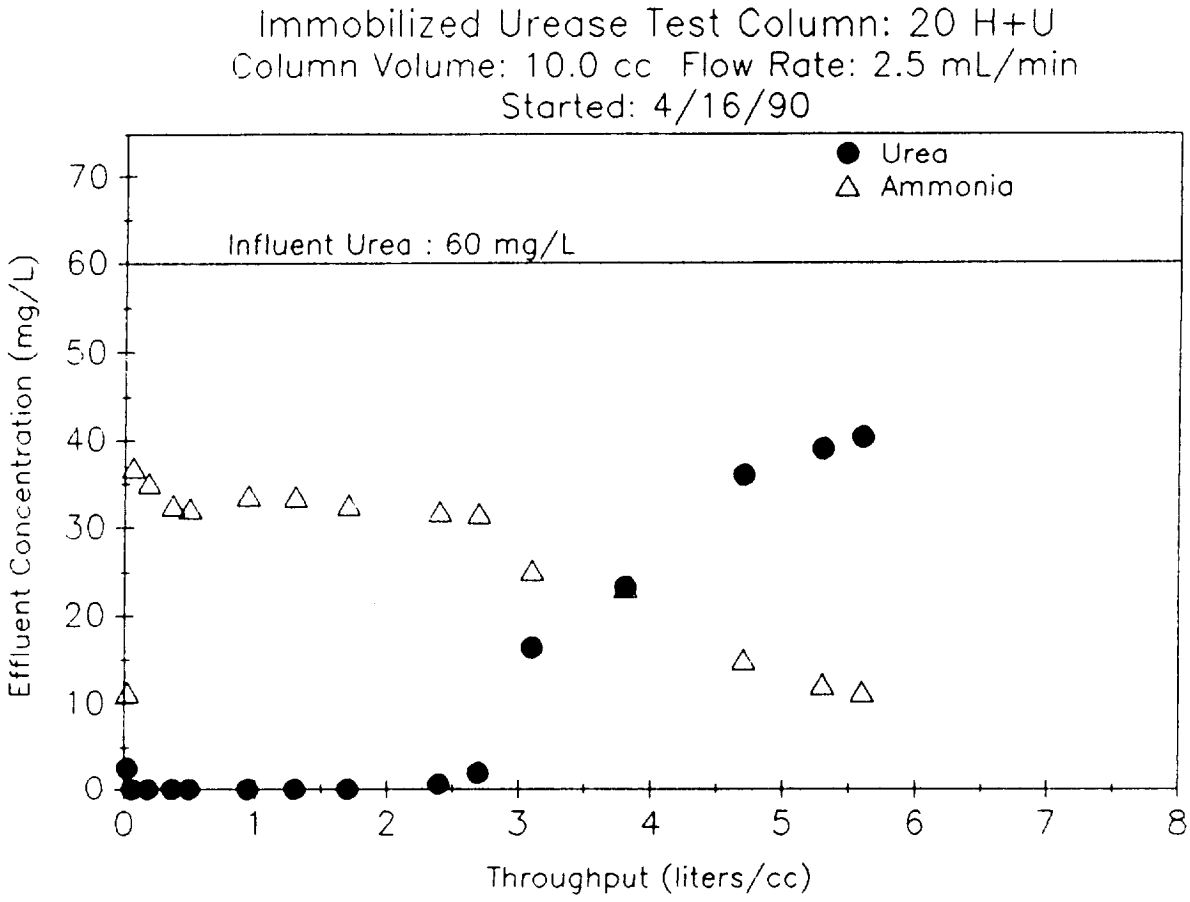

Immobilized Urease Test Column: B/N 900206-1
Column Volume: 18.7 cc Flow Rate: 2.5 mL/min
Started: 2/6/90
Urea
Ammonia
Influent Urea: 60 mg/L
Effluent Concentration (mg/L)
Throughput (liters/cc)
0
10
20
30
40
50
60
70
80
0
1
2
3
4
5
6

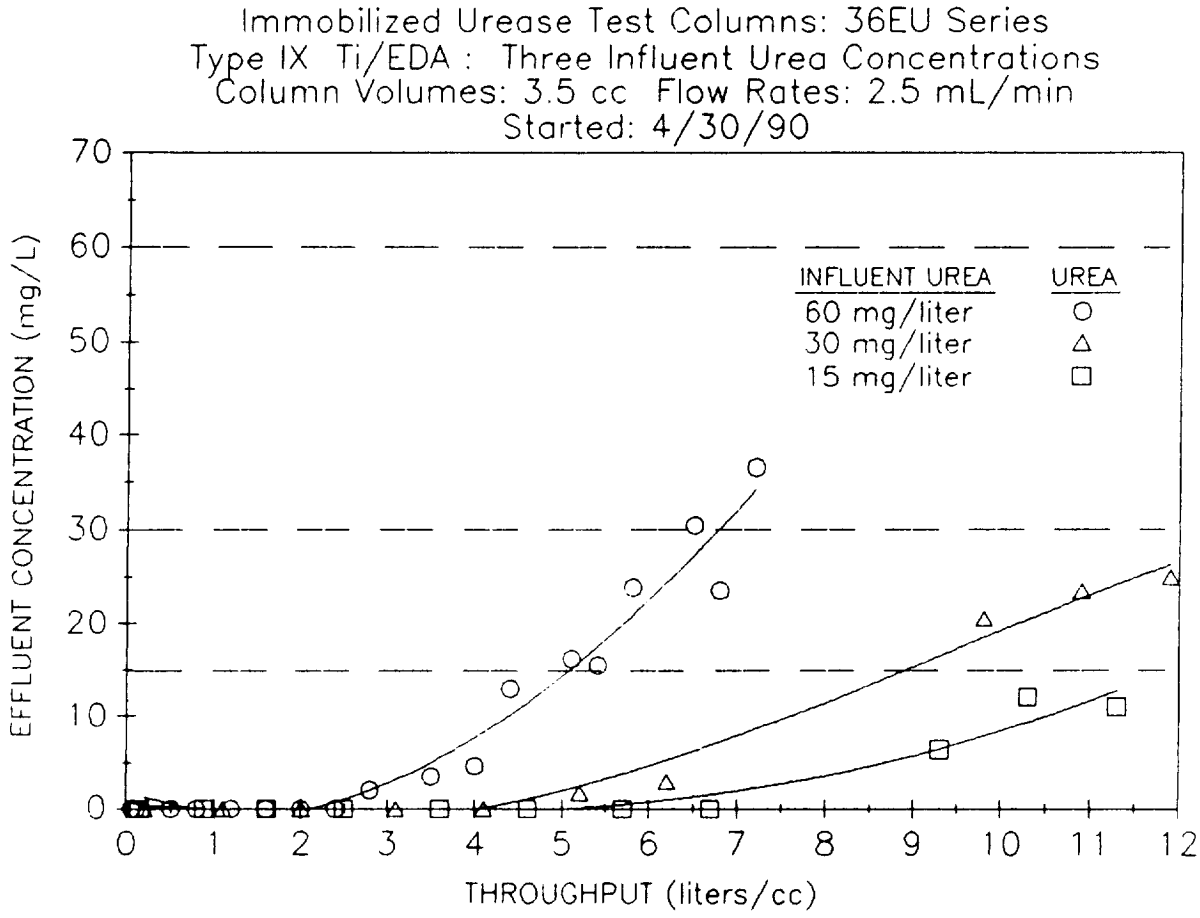

Immobilized Urease Test Column: B/N 900228-1
Column Volume: 5.0 cc Flow Rate: Varied
Started: 2/28/90
Urea
Ammonia
Influent Urea: 60 mg/L
3.0 ml/min
2.5 ml/min
0.5 ml/min
Effluent Concentration (mg/L)
Throughput (liters/cc)
0
10
20
30
40
50
60
70
80
0
1
2
3
4
5
6
7
8
9
10
11
12

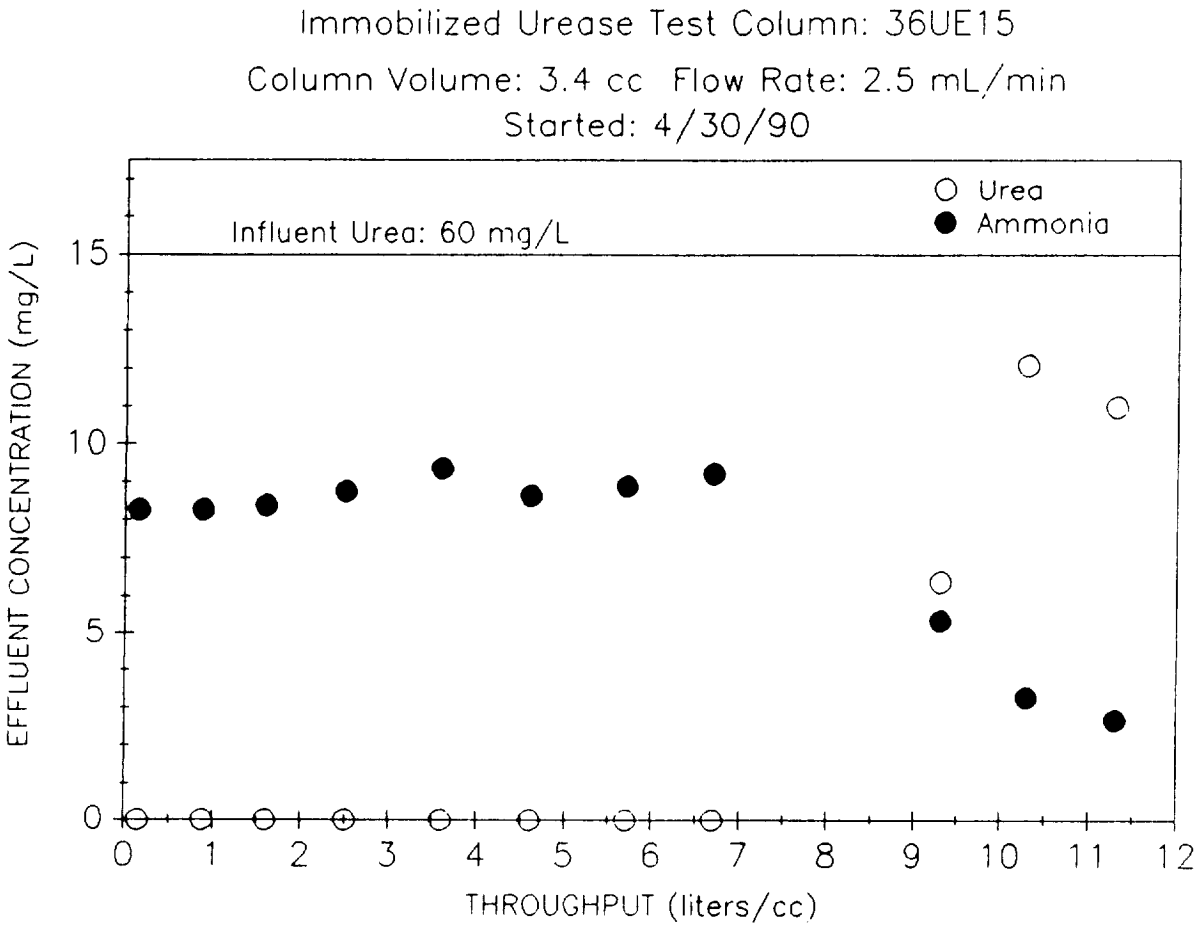

Immobilized Urease Test Column: Type C-3
Column Volume: 9.2 cc Flow Rate: 4.5 mL/min
Started: 1/90
Urea
Ammonia
Influent Urea: 60 mg/L
Effluent Concentration (mg/L)
Throughput (liters/cc)
0
10
20
30
40
50
60
70
80
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0

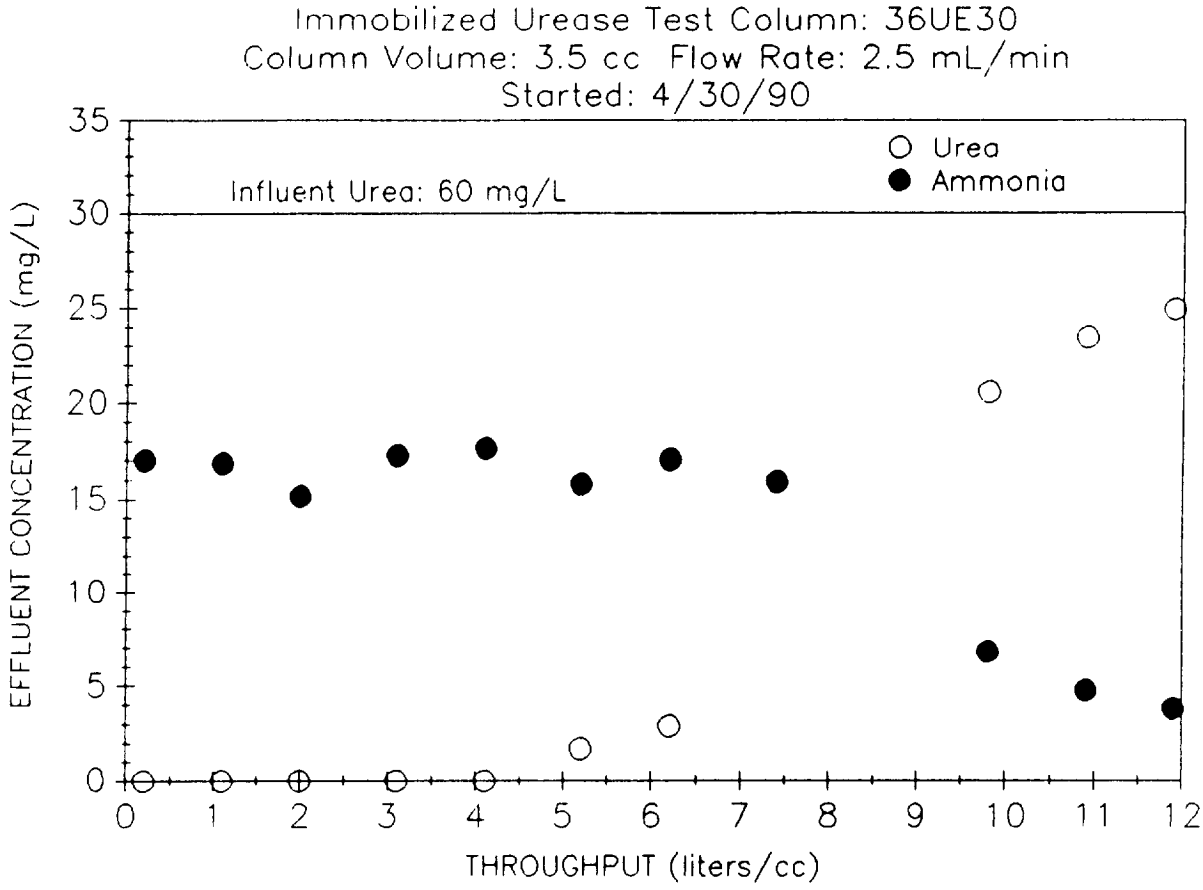

Immobilized Urease Test Column: B/N 900130
Column Volume: 4.6 cc Flow Rate: 2.5 mL/min
Started: 1/30/90
Urea
Ammonia
Influent Urea: 60 mg/L
Effluent Concentration (mg/L)
Throughput (liters/cc)
0
10
20
30
40
50
60
70
80
90

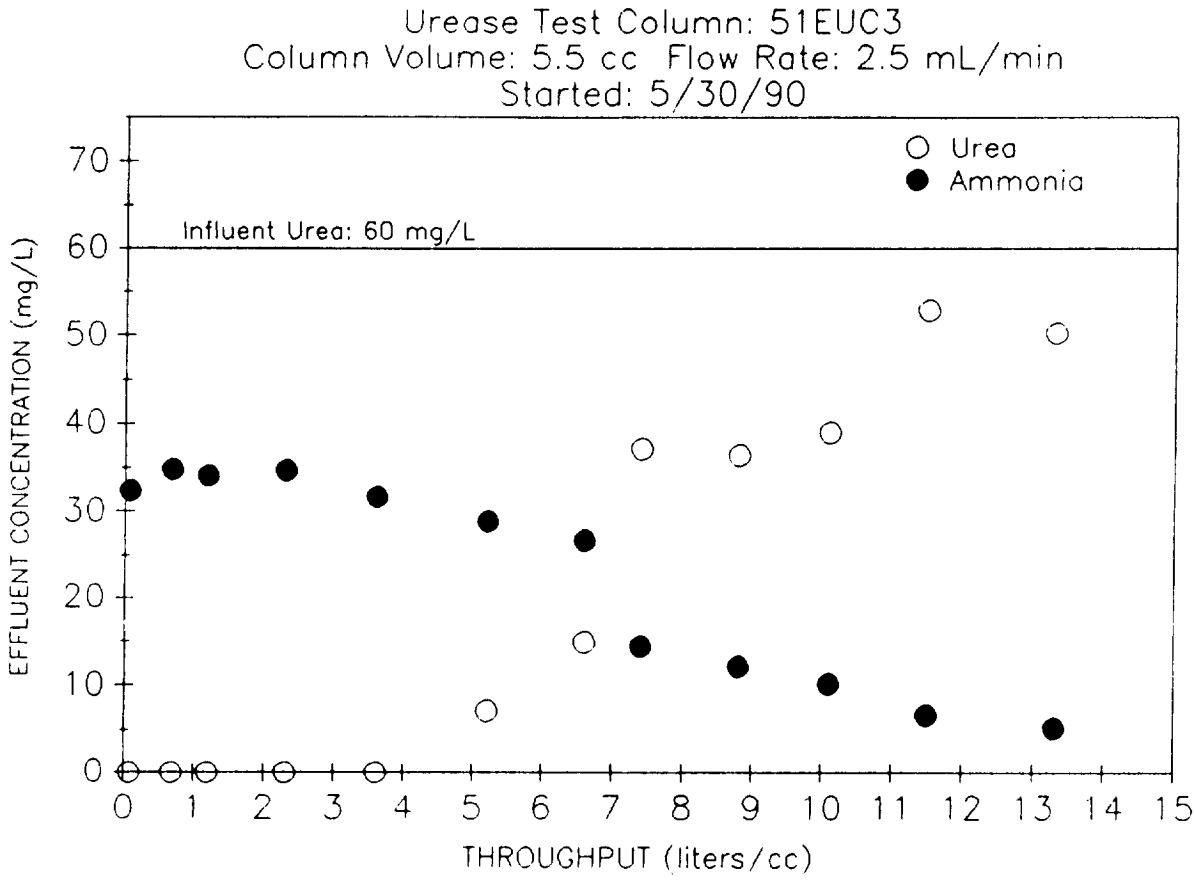

Immobilized Urease Test Column: 13EU
Column Volume: 10.0 cc Flow Rate: 2.5 mL/min
Started: 4/3/90
Influent Urea: 60 mg/L
UREA
AMMONIA
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters/cc)
0
10
20
30
40
50
60
70
0
1
2
3
4
5
6
7
8

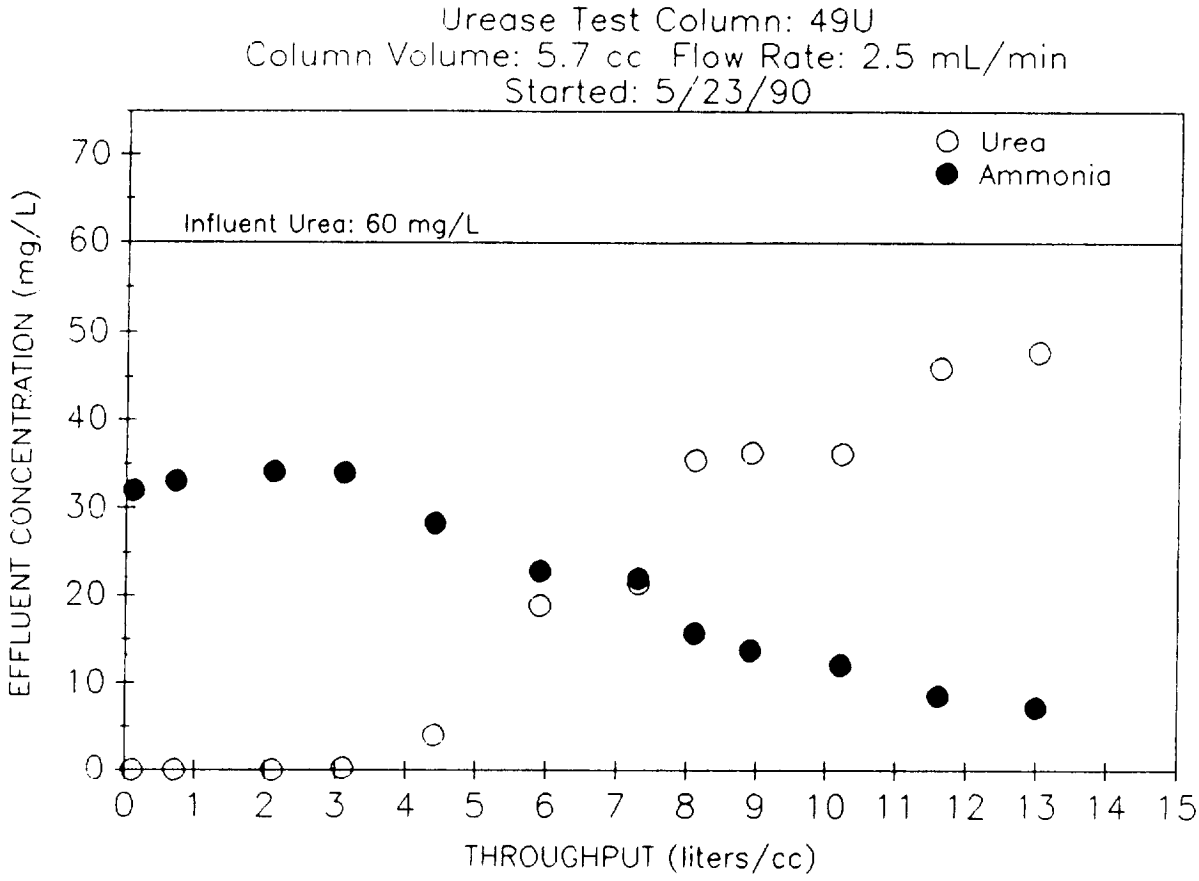

Immobilized Urease Test Column: 20 H+U
Column Volume: 10.0 cc Flow Rate: 2.5 mL/min
Started: 4/16/90
Urea
Ammonia
Influent Urea : 60 mg/L
Effluent Concentration (mg/L)
70
60
50
40
30
20
10
0
0
1
2
3
4
5
6
7
8
Throughput (liters/cc)

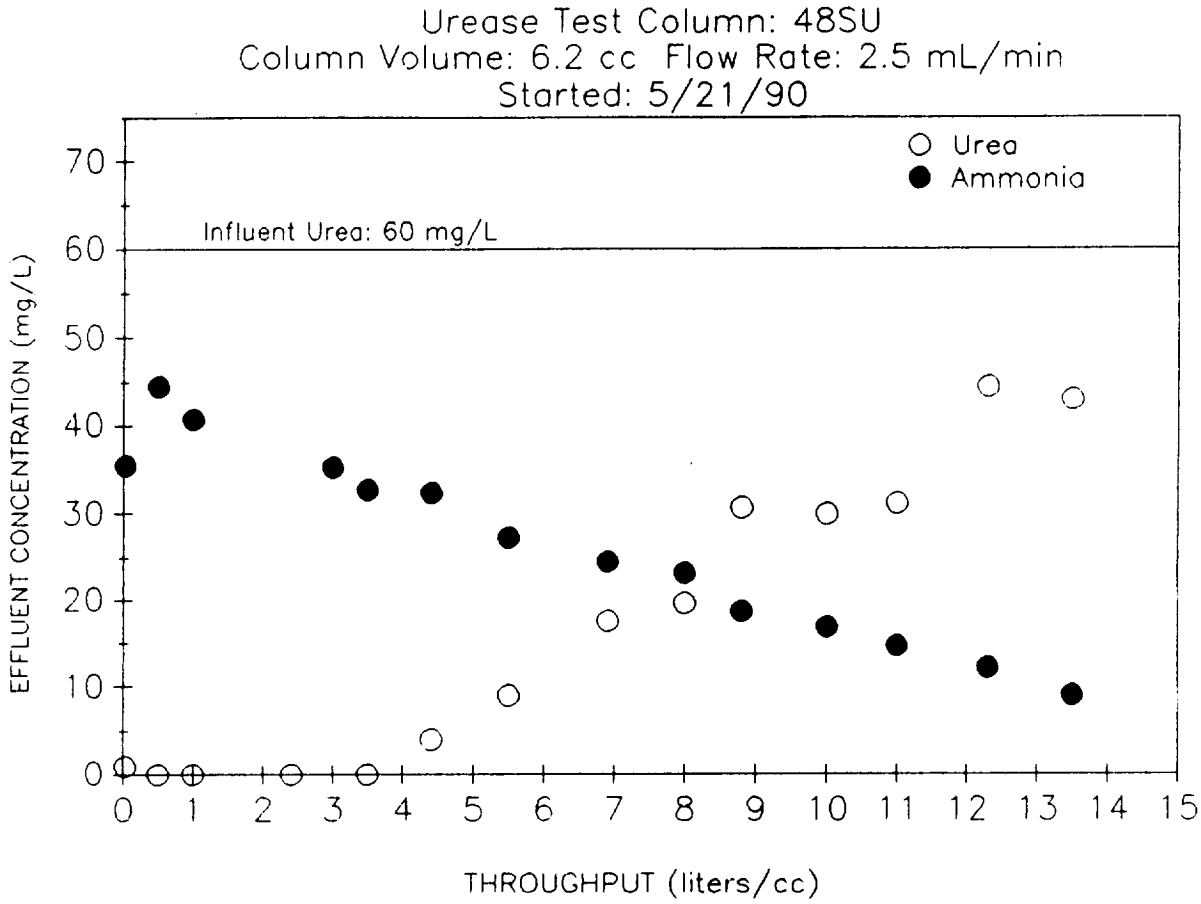

Immobilized Urease Test Columns: 36EU Series
Type IX Ti/EDA : Three Influent Urea Concentrations
Column Volumes: 3.5 cc Flow Rates: 2.5 mL/min
Started: 4/30/90
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters/cc)
INFLUENT UREA
UREA
60 mg/liter
30 mg/liter
15 mg/liter
0
10
20
30
40
50
60
70
0
1
2
3
4
5
6
7
8
9
10
11
12

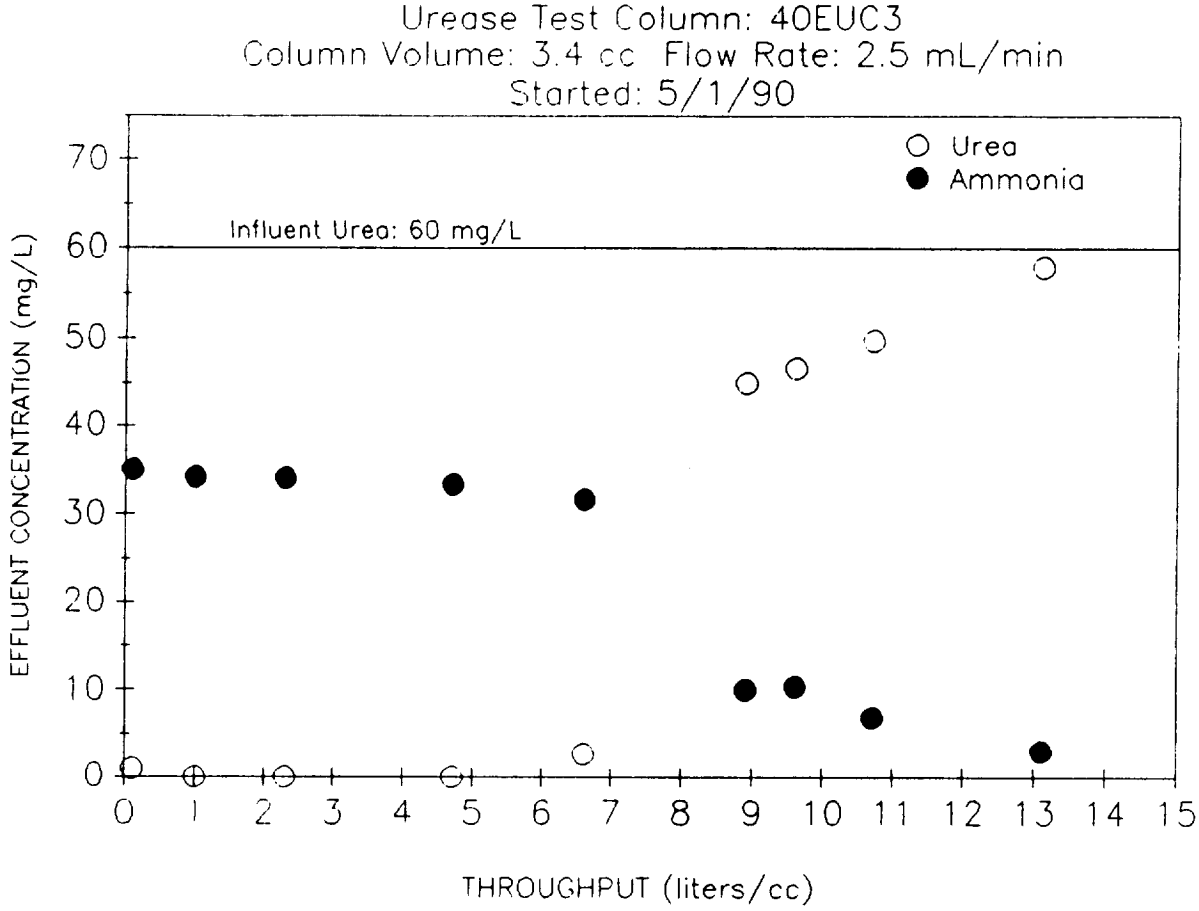

Immobilized Urease Test Column: 36UE15
Column Volume: 3.4 cc Flow Rate: 2.5 mL/min
Started: 4/30/90
Influent Urea: 60 mg/L
Urea
Ammonia
EFFLUENT CONCENTRATION (mg/L)
0
5
10
15
0 1 2 3 4 5 6 7 8 9 10 11 12
THROUGHPUT (liters/cc)

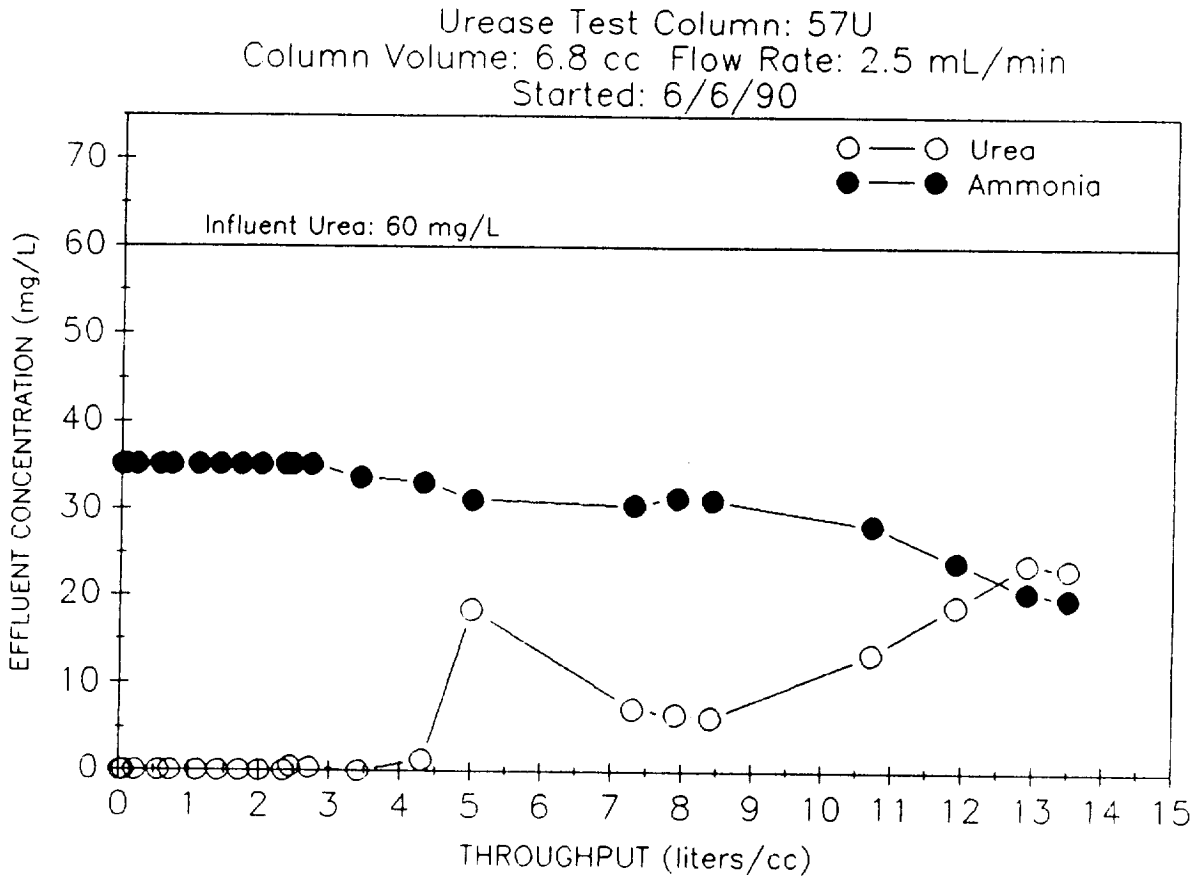

Immobilized Urease Test Column: 36UE30
Column Volume: 3.5 cc Flow Rate: 2.5 mL/min
Started: 4/30/90
Influent Urea: 60 mg/L
Urea
Ammonia
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters/cc)
0
5
10
15
20
25
30
35
0
1
2
3
4
5
6
7
8
9
10
11
12

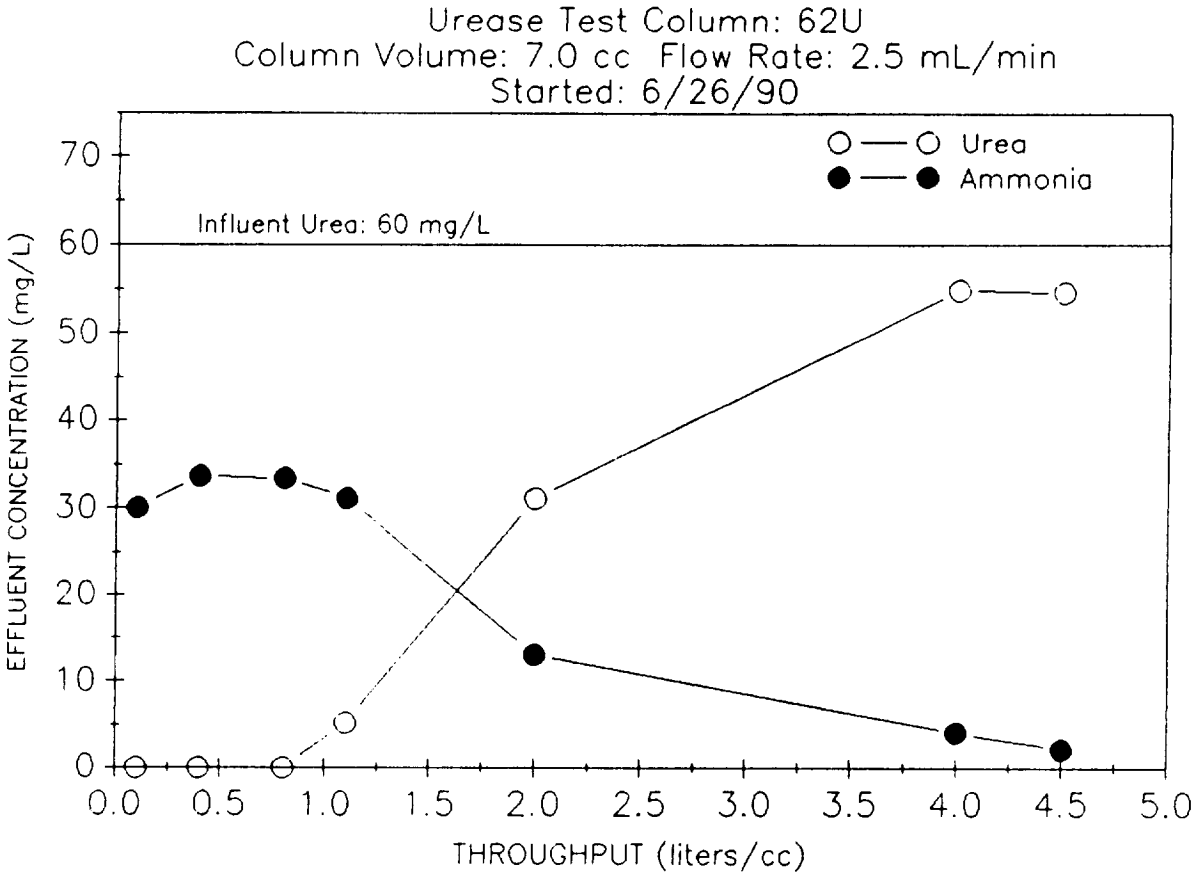

Urease Test Column: 51EUC3
Column Volume: 5.5 cc Flow Rate: 2.5 mL/min
Started: 5/30/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters/cc)
0
10
20
30
40
50
60
70
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

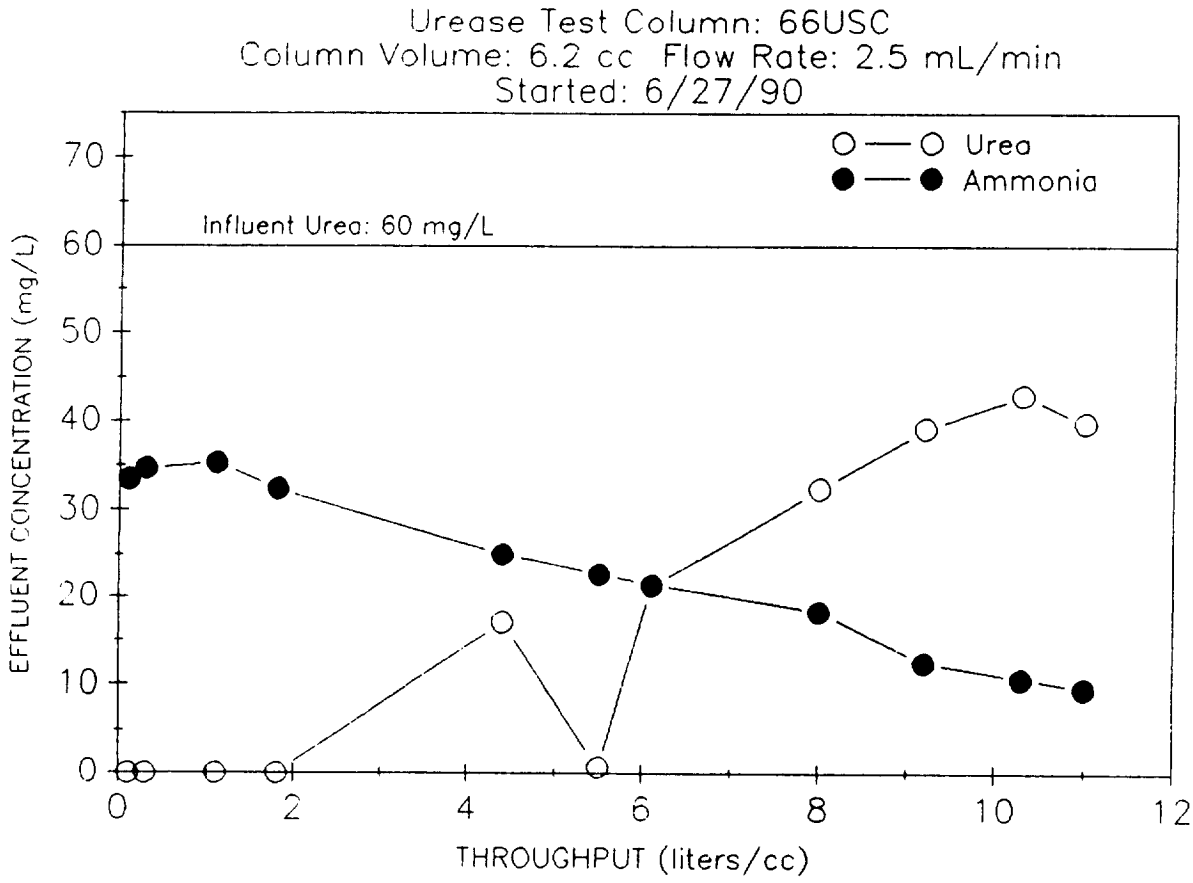

Urease Test Column: 49U
Column Volume: 5.7 cc Flow Rate: 2.5 mL/min
Started: 5/23/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters/cc)
0
10
20
30
40
50
60
70
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15

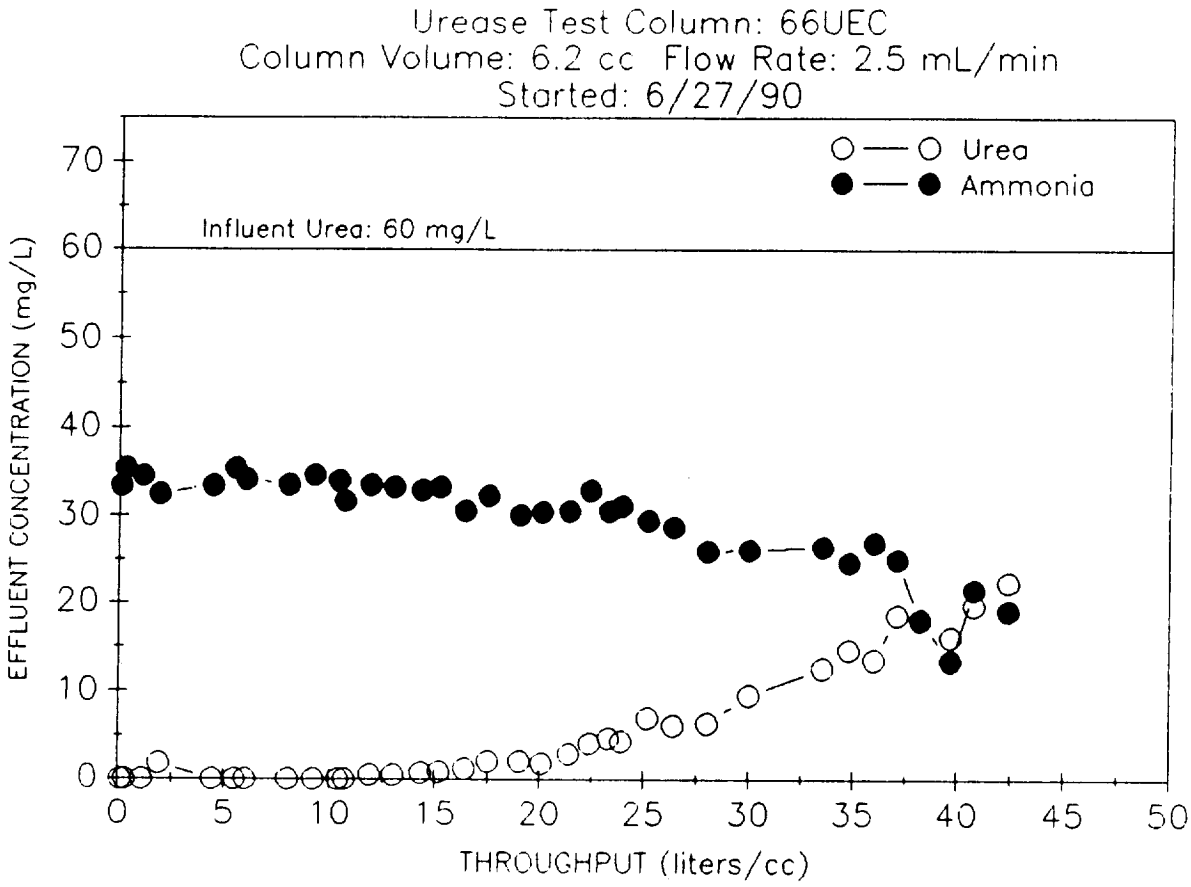

Urease Test Column: 48SU
Column Volume: 6.2 cc Flow Rate: 2.5 mL/min
Started: 5/21/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
0
10
20
30
40
50
60
70
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15
THROUGHPUT (liters/cc)

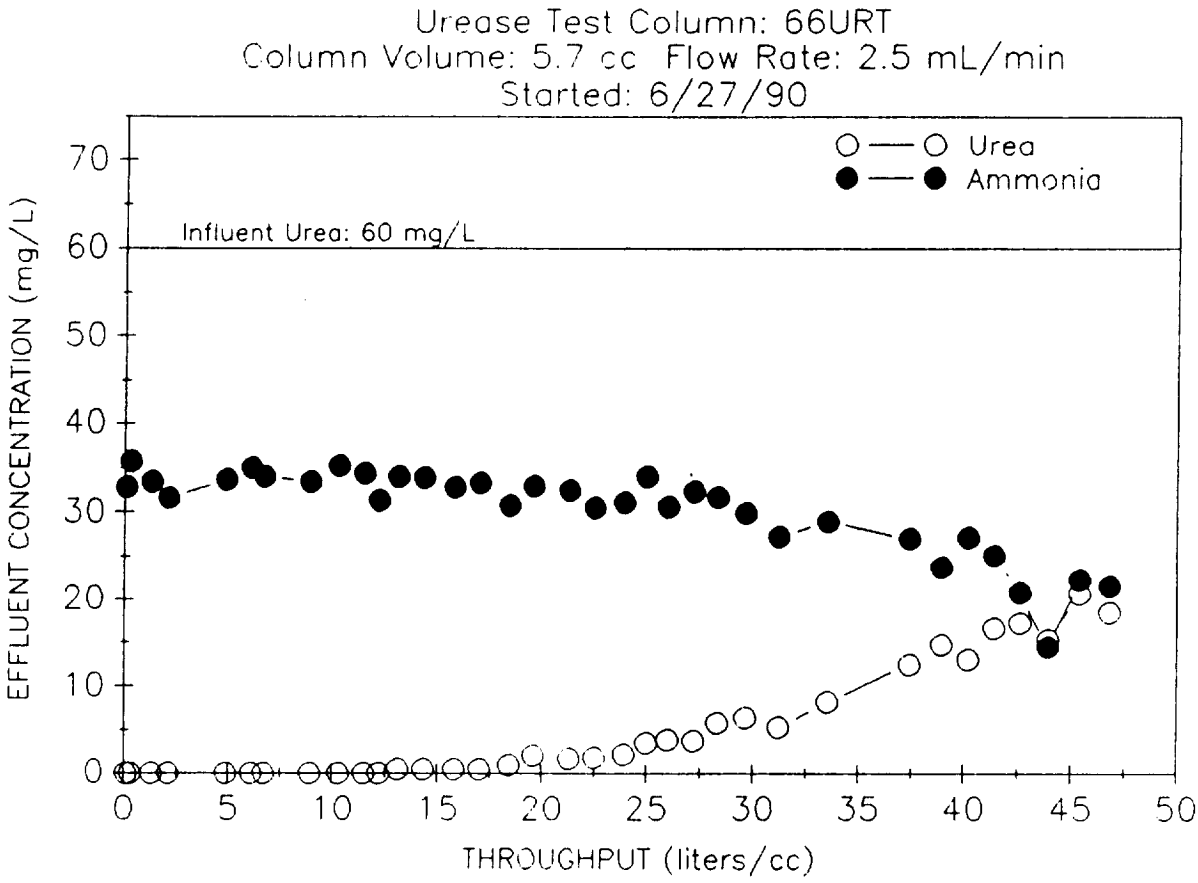

Urease Test Column: 40EUC3
Column Volume: 3.4 cc Flow Rate: 2.5 mL/min
Started: 5/1/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
0
10
20
30
40
50
60
70
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15
THROUGHPUT (liters/cc)

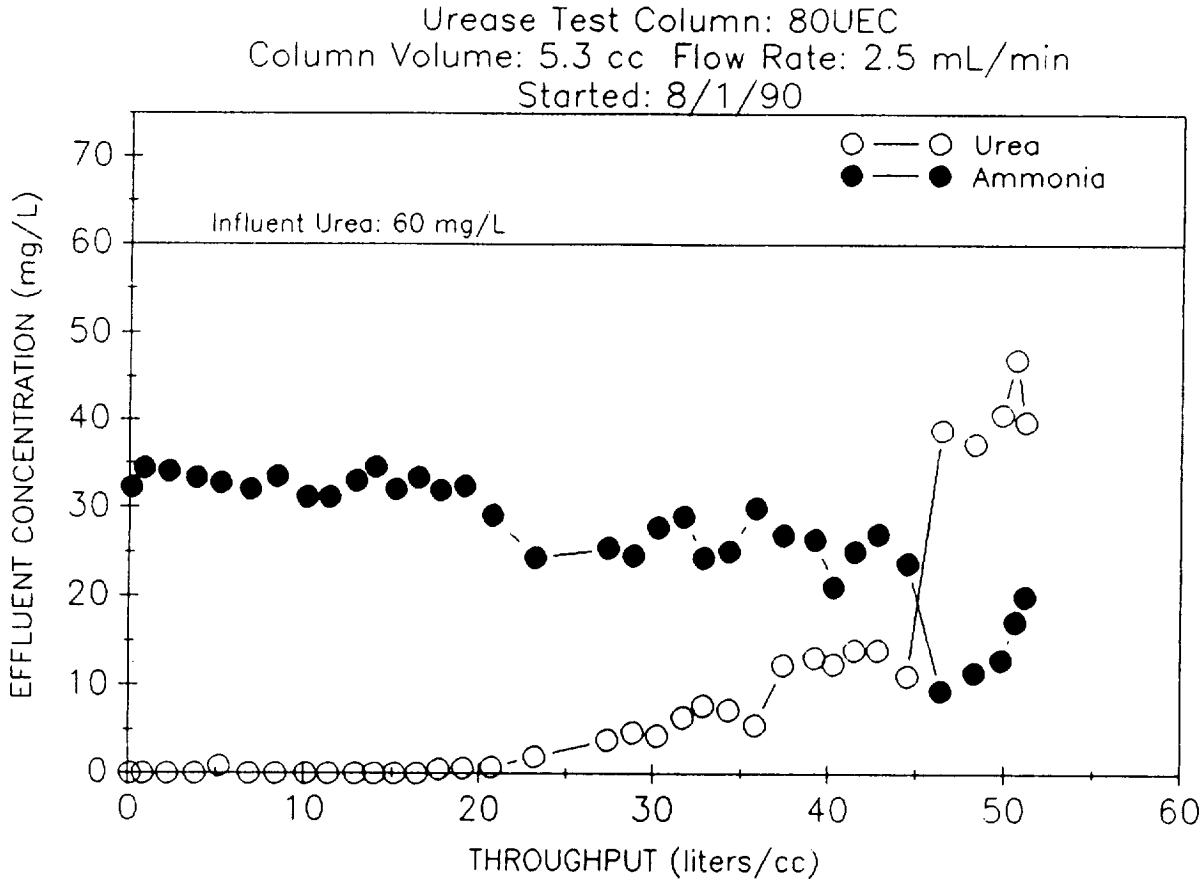

Urease Test Column: 57U
Column Volume: 6.8 cc Flow Rate: 2.5 mL/min
Started: 6/6/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
0
10
20
30
40
50
60
70
0 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15
THROUGHPUT (liters/cc)

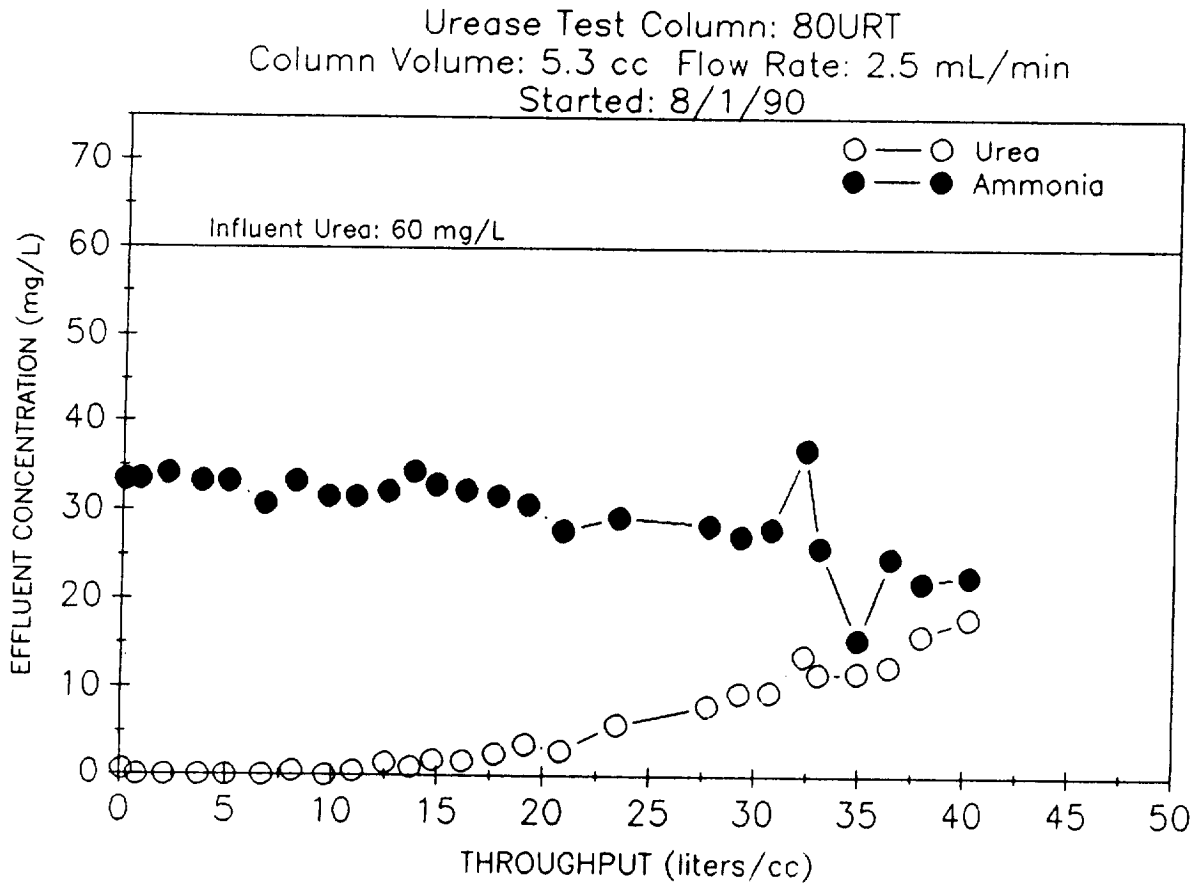

Urease Test Column: 62U
Column Volume: 7.0 cc Flow Rate: 2.5 mL/min
Started: 6/26/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
THROUGHPUT (liters/cc)

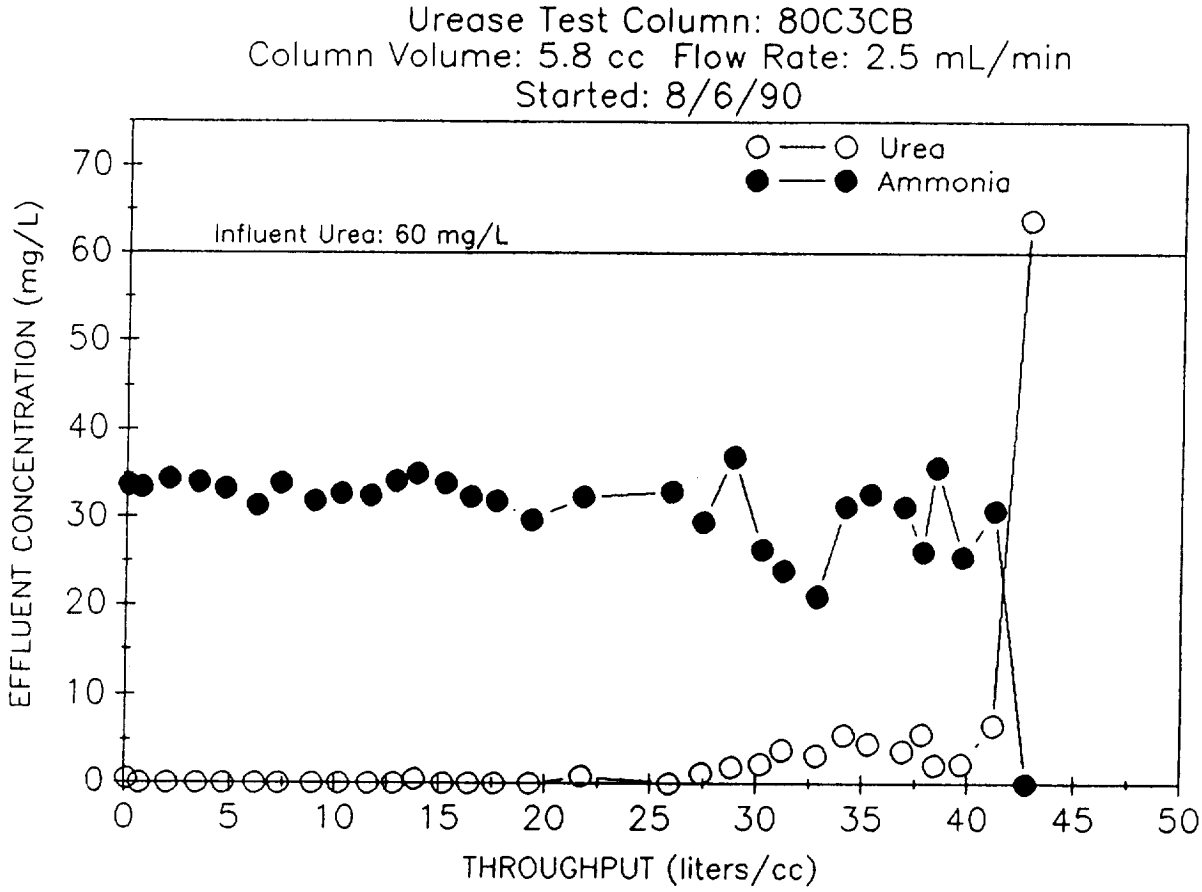

Urease Test Column: 66USC
Column Volume: 6.2 cc Flow Rate: 2.5 mL/min
Started: 6/27/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0
2
4
6
8
10
12
THROUGHPUT (liters/cc)

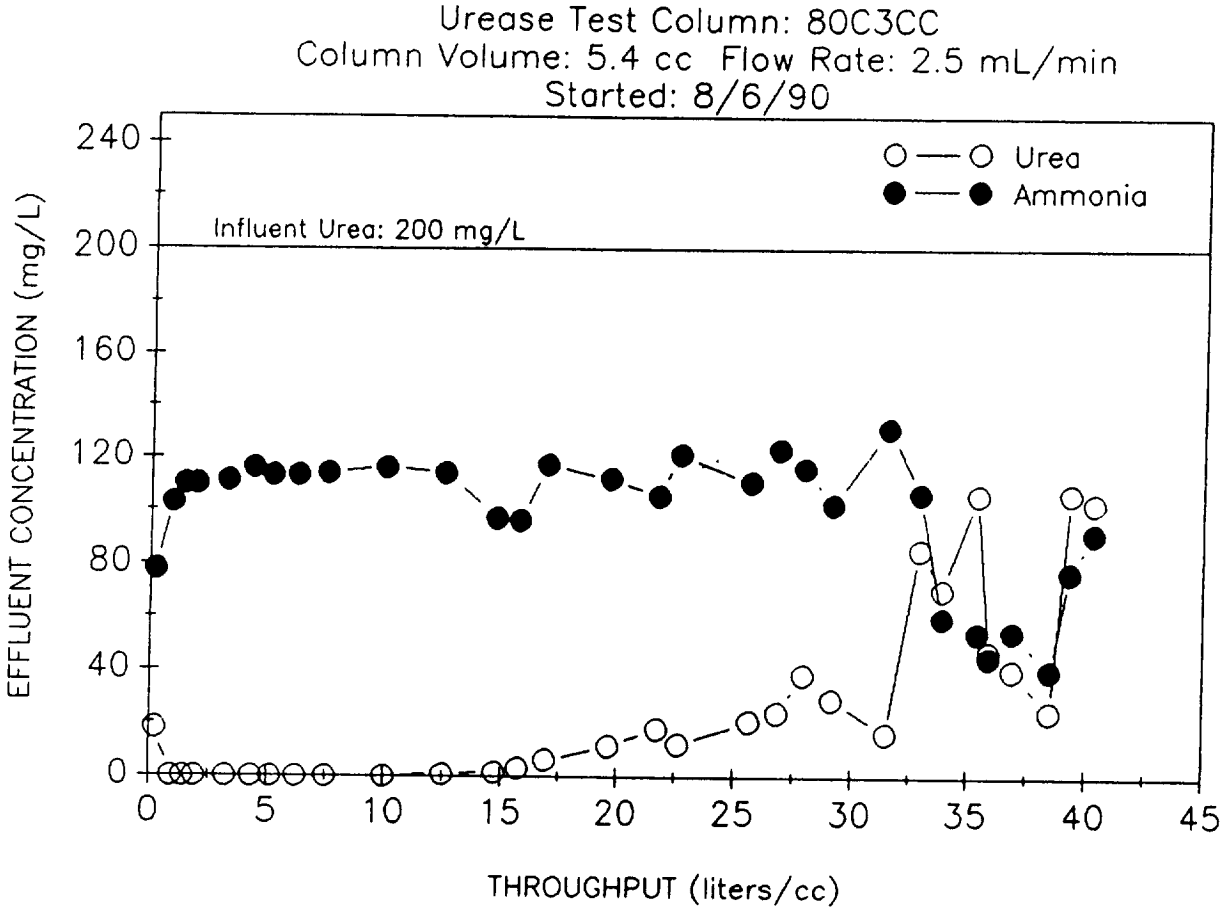

Urease Test Column: 66UEC
Column Volume: 6.2 cc Flow Rate: 2.5 mL/min
Started: 6/27/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters/cc)
0
10
20
30
40
50
60
70
0
5
10
15
20
25
30
35
40
45
50

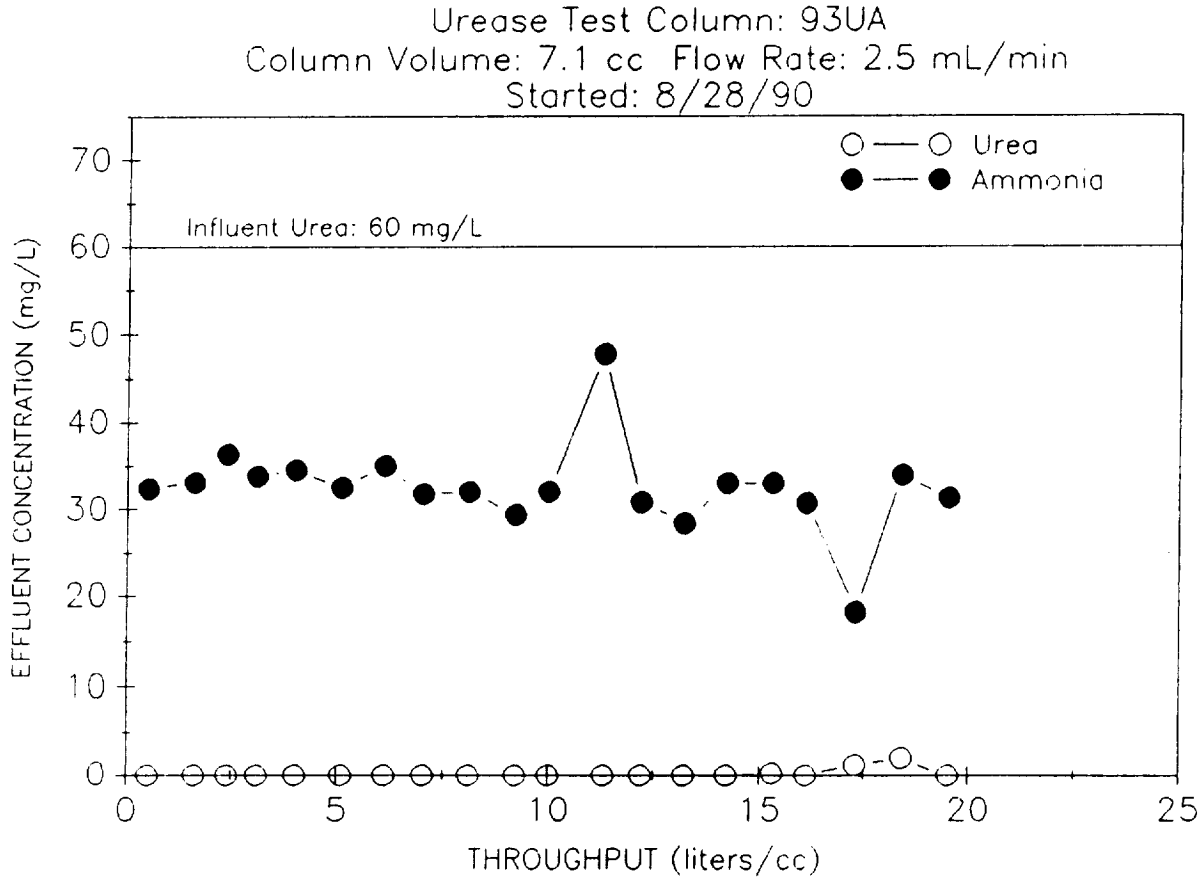

Urease Test Column: 66URT
Column Volume: 5.7 cc Flow Rate: 2.5 mL/min
Started: 6/27/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters/cc)
0
10
20
30
40
50
60
70
0
5
10
15
20
25
30
35
40
45
50

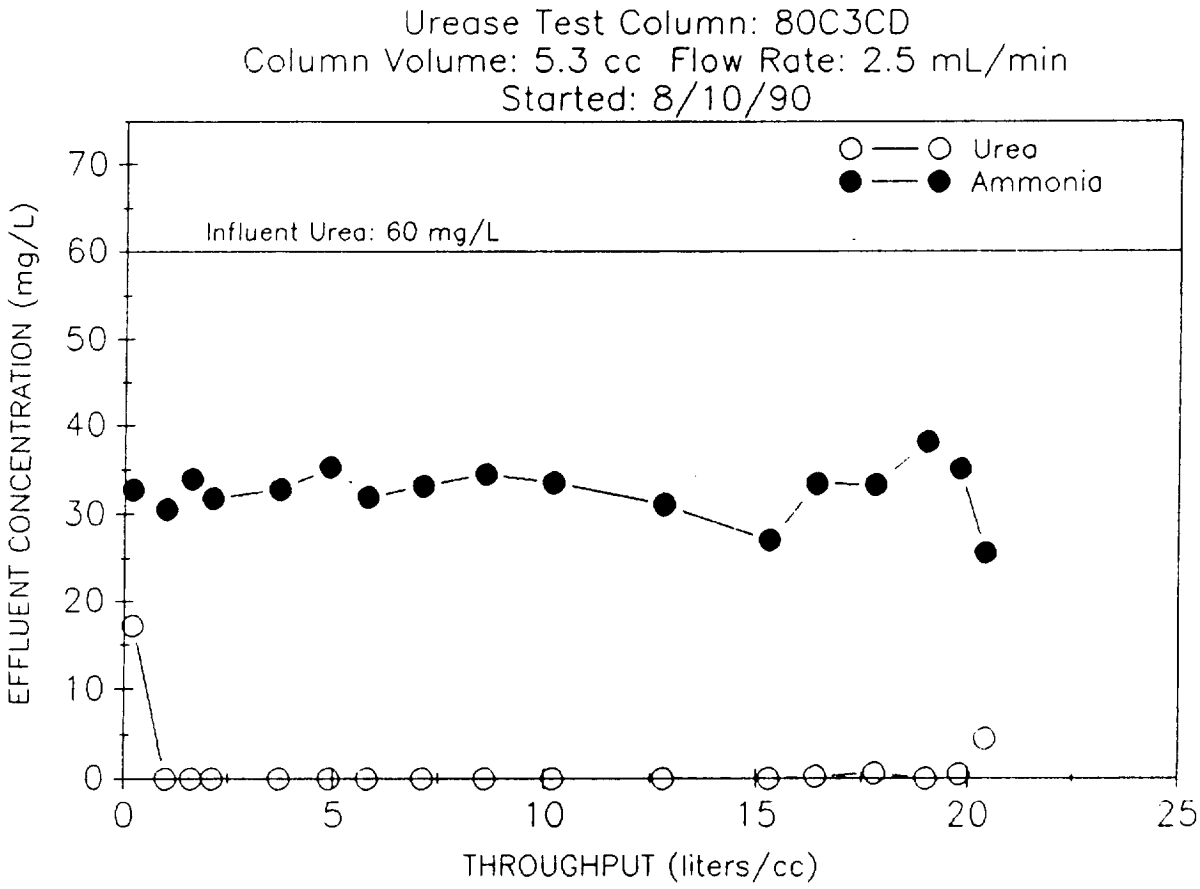

Urease Test Column: 80UEC
Column Volume: 5.3 cc Flow Rate: 2.5 mL/min
Started: 8/1/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters/cc)
0
10
20
30
40
50
60
70

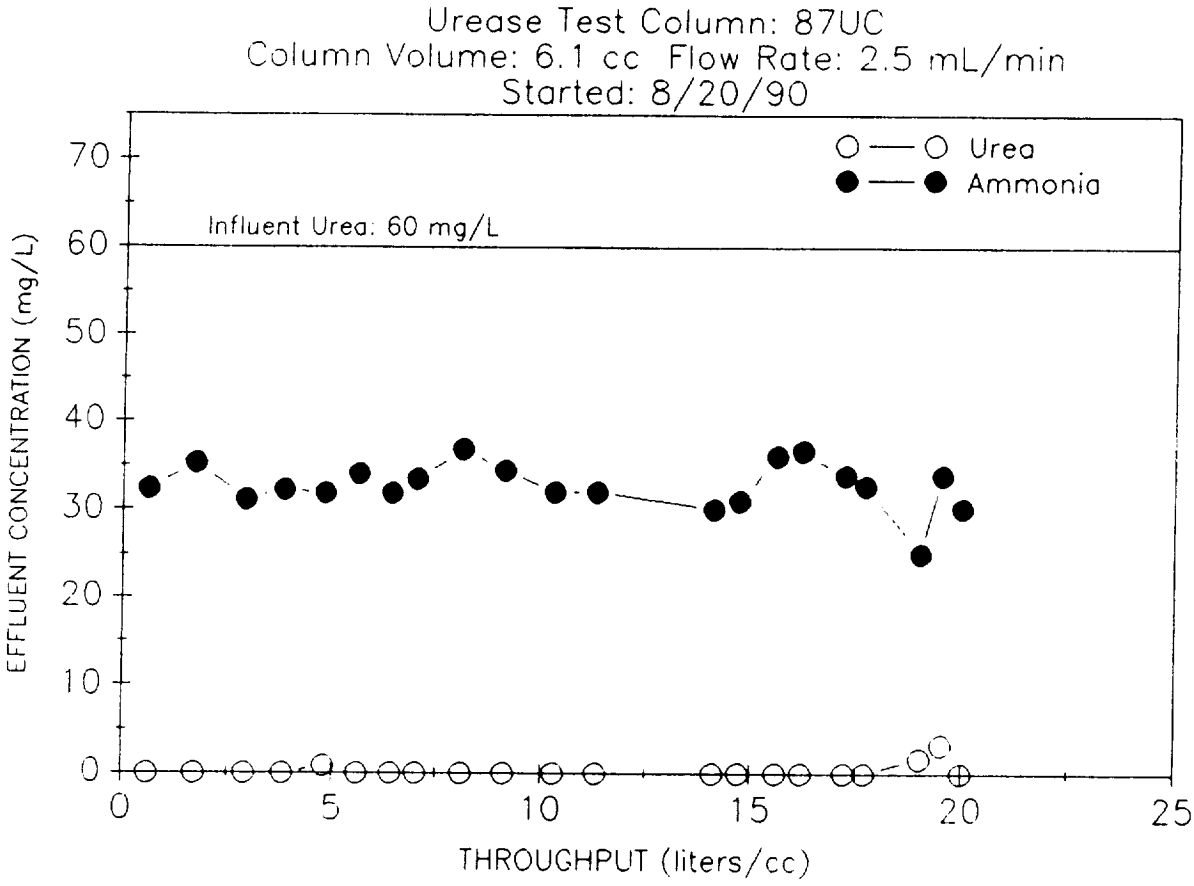

Urease Test Column: 80URT
Column Volume: 5.3 cc Flow Rate: 2.5 mL/min
Started: 8/1/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters/cc)
0
10
20
30
40
50
60
70
0
5
10
15
20
25
30
35
40
45
50

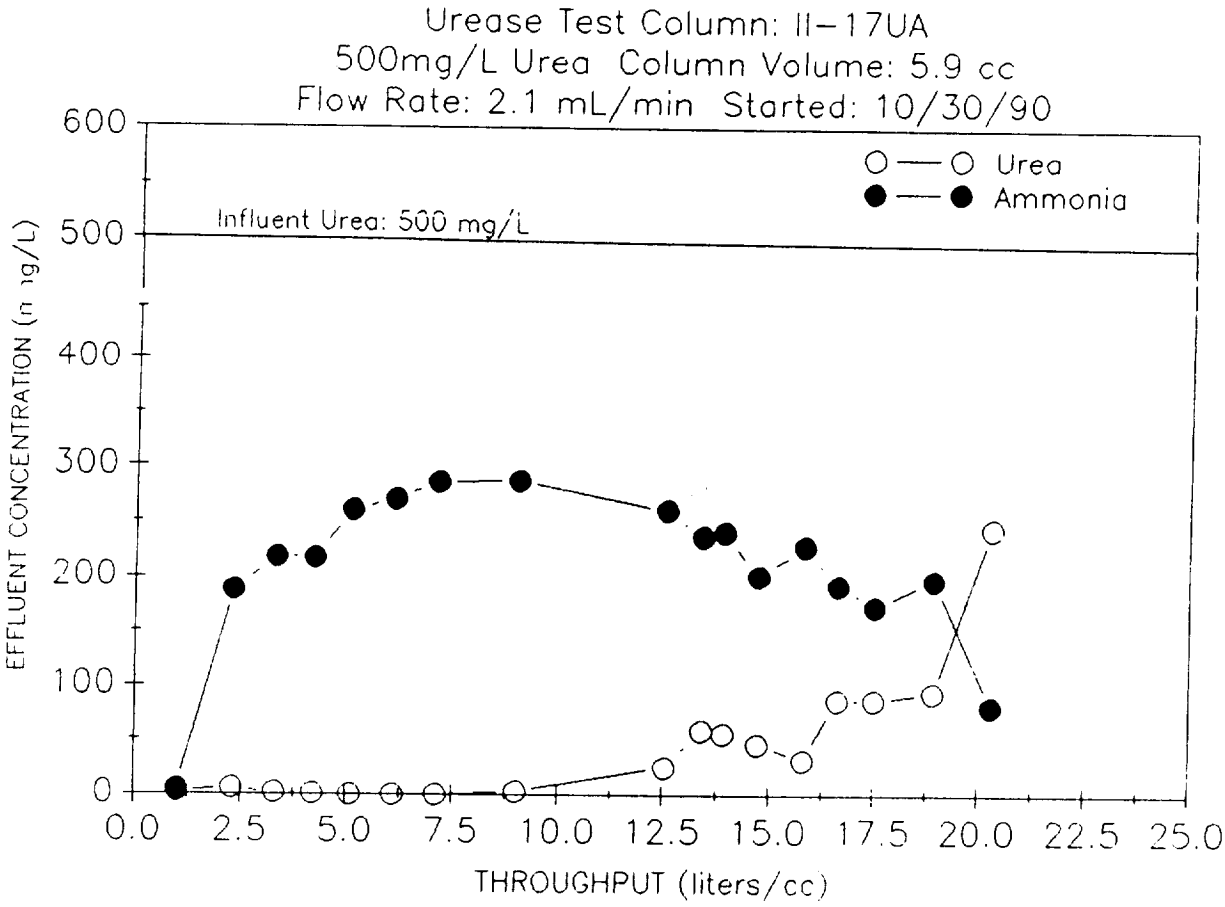

Urease Test Column: 80C3CB
Column Volume: 5.8 cc Flow Rate: 2.5 mL/min
Started: 8/6/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
0
10
20
30
40
50
60
70
0
5
10
15
20
25
30
35
40
45
50
THROUGHPUT (liters/cc)

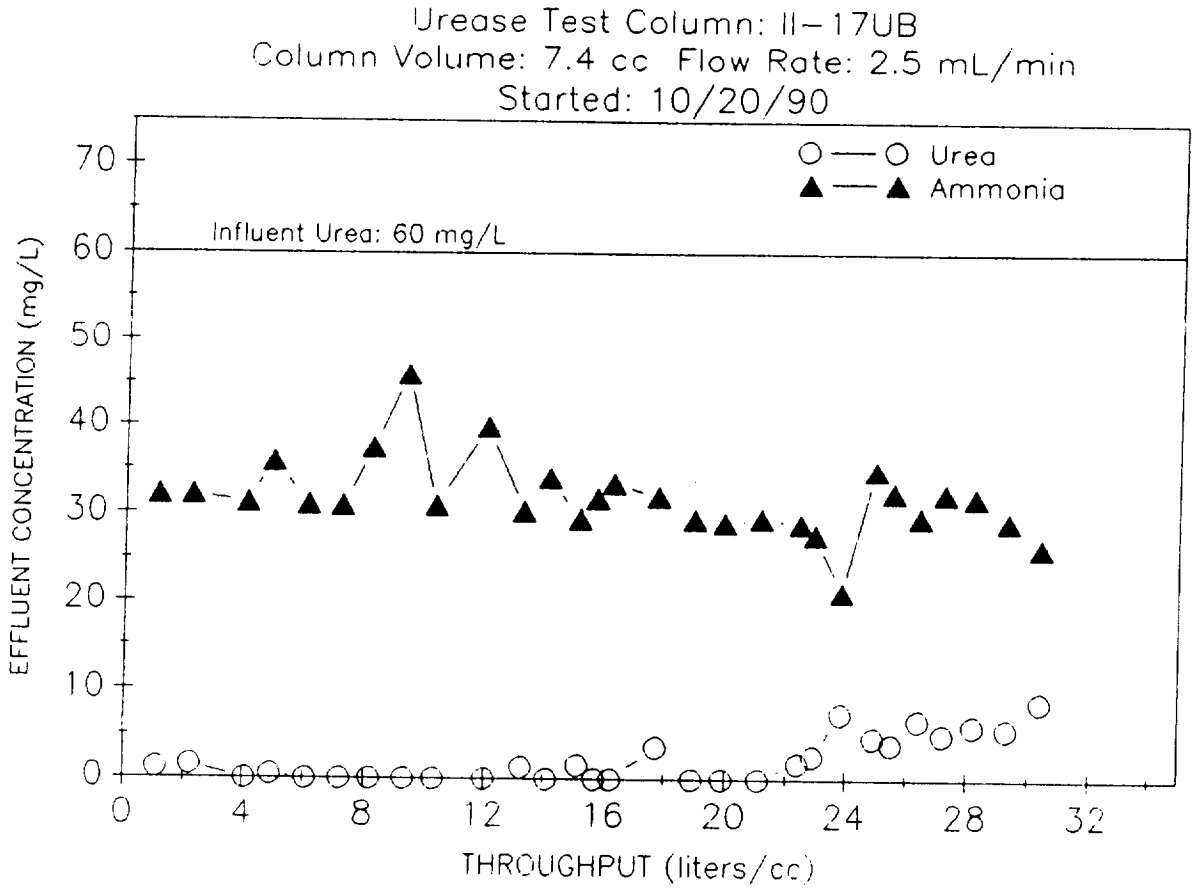

Urease Test Column: 80C3CC
Column Volume: 5.4 cc Flow Rate: 2.5 mL/min
Started: 8/6/90
Urea
Ammonia
Influent Urea: 200 mg/L
EFFLUENT CONCENTRATION (mg/L)
240
200
160
120
80
40
0
0
5
10
15
20
25
30
35
40
45
THROUGHPUT (liters/cc)

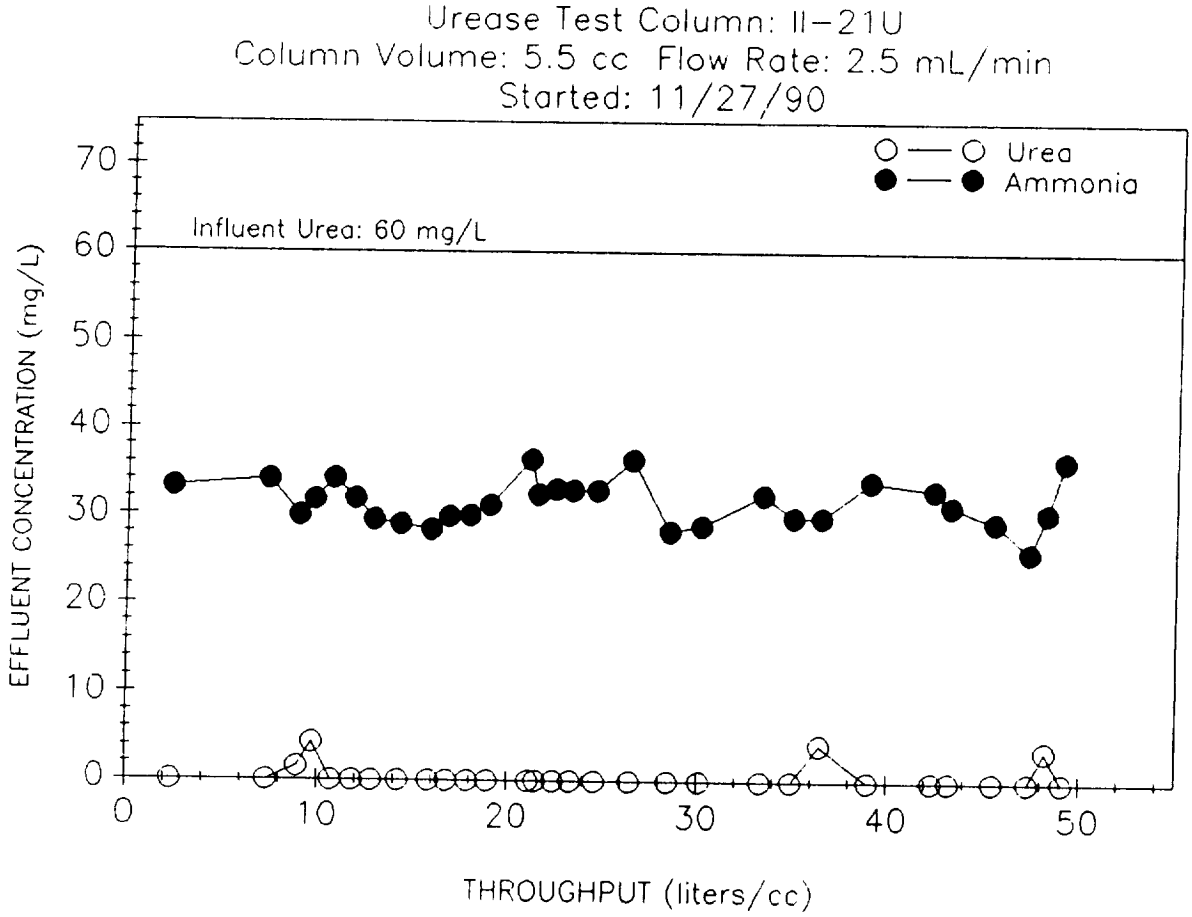

Urease Test Column: 93UA
Column Volume: 7.1 cc Flow Rate: 2.5 mL/min
Started: 8/28/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0
5
10
15
20
25
THROUGHPUT (liters/cc)

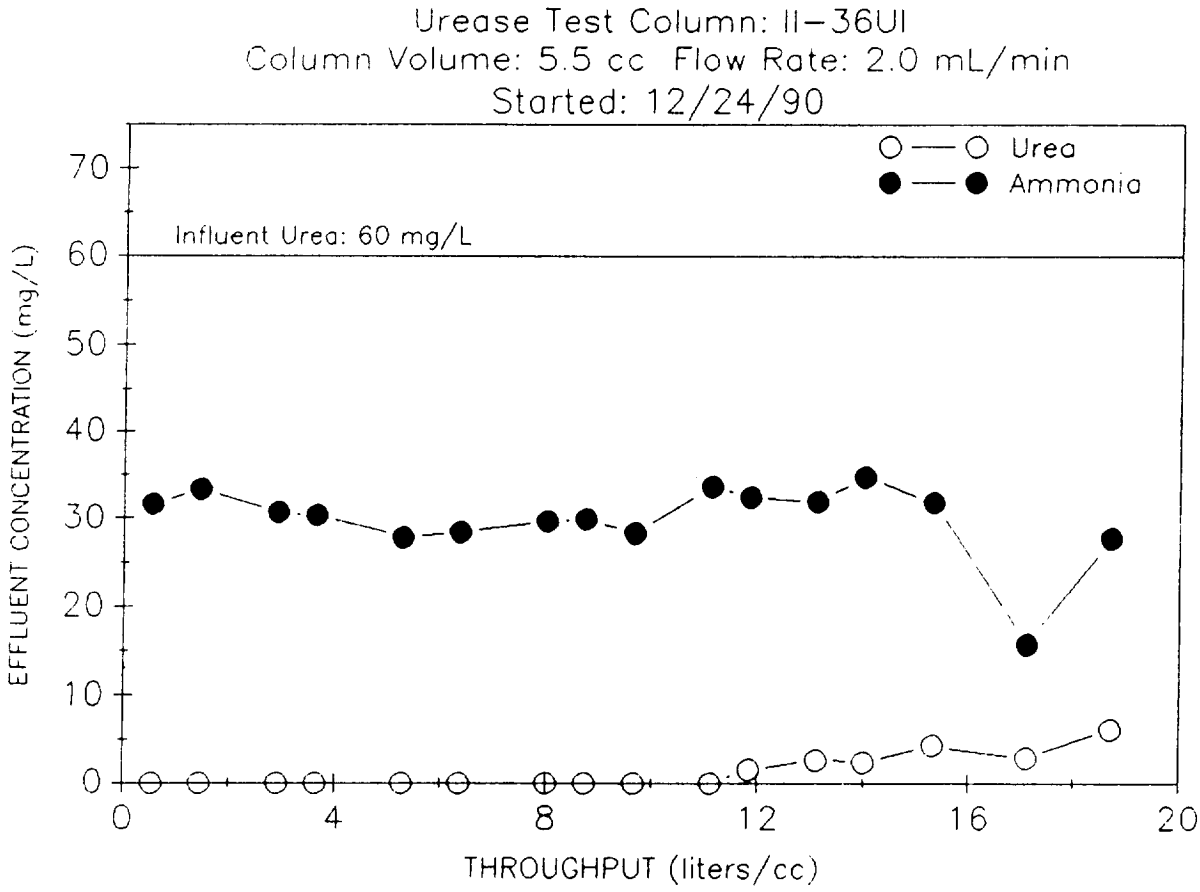

Urease Test Column: 80C3CD
Column Volume: 5.3 cc Flow Rate: 2.5 mL/min
Started: 8/10/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0
5
10
15
20
25
THROUGHPUT (liters/cc)

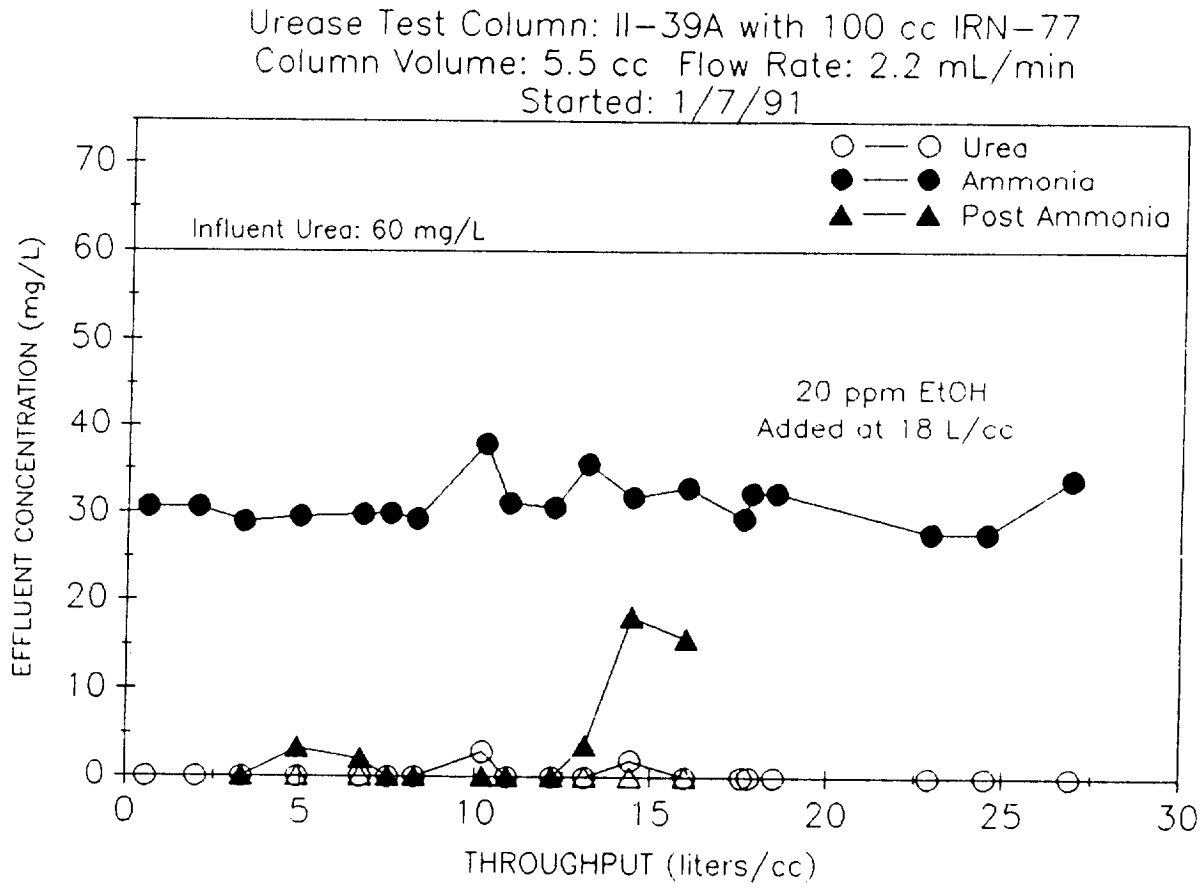

Urease Test Column: 87UC
Column Volume: 6.1 cc Flow Rate: 2.5 mL/min
Started: 8/20/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0
5
10
15
20
25
THROUGHPUT (liters/cc)

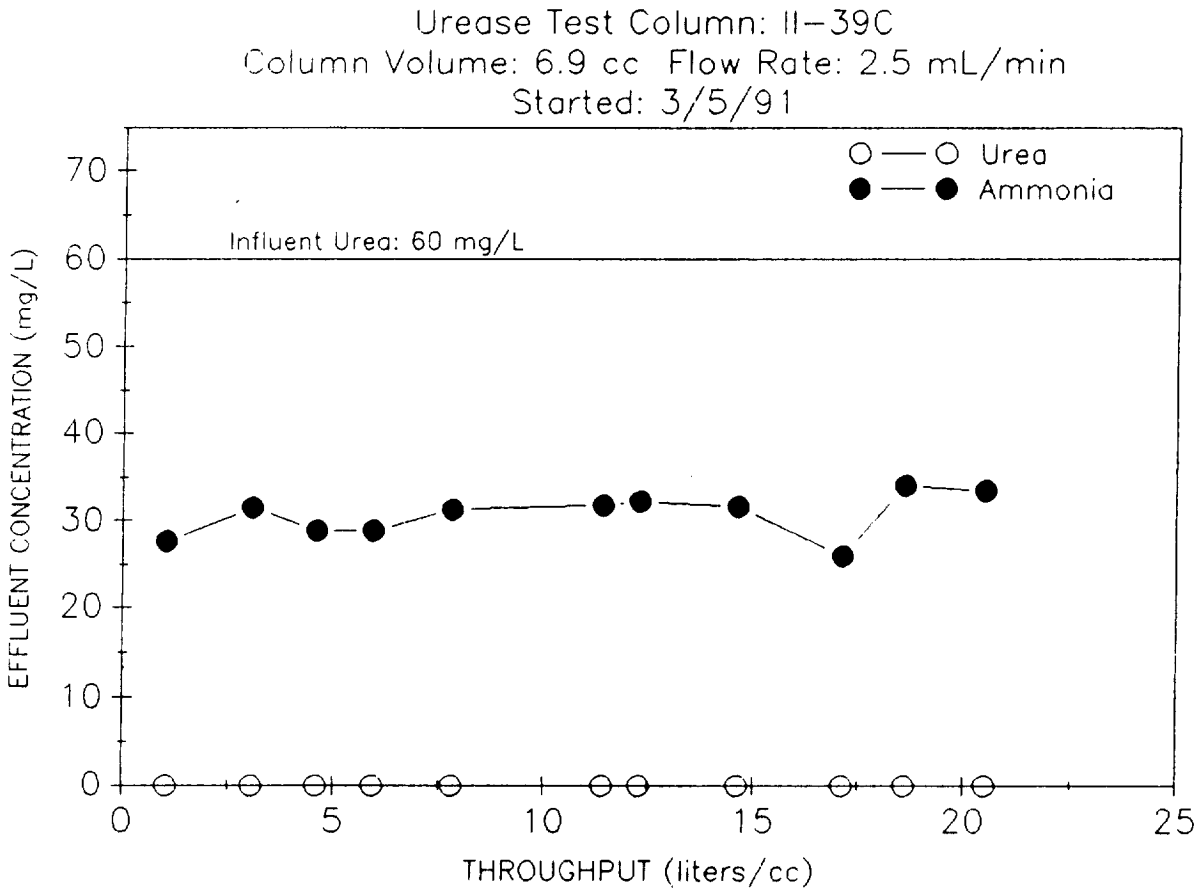

Urease Test Column: II-17UA
500mg/L Urea Column Volume: 5.9 cc
Flow Rate: 2.1 mL/min Started: 10/30/90
Urea
Ammonia
Influent Urea: 500 mg/L
EFFLUENT CONCENTRATION (mg/L)
600
500
400
300
200
100
0
0.0
2.5
5.0
7.5
10.0
12.5
15.0
17.5
20.0
22.5
25.0
THROUGHPUT (liters/cc)

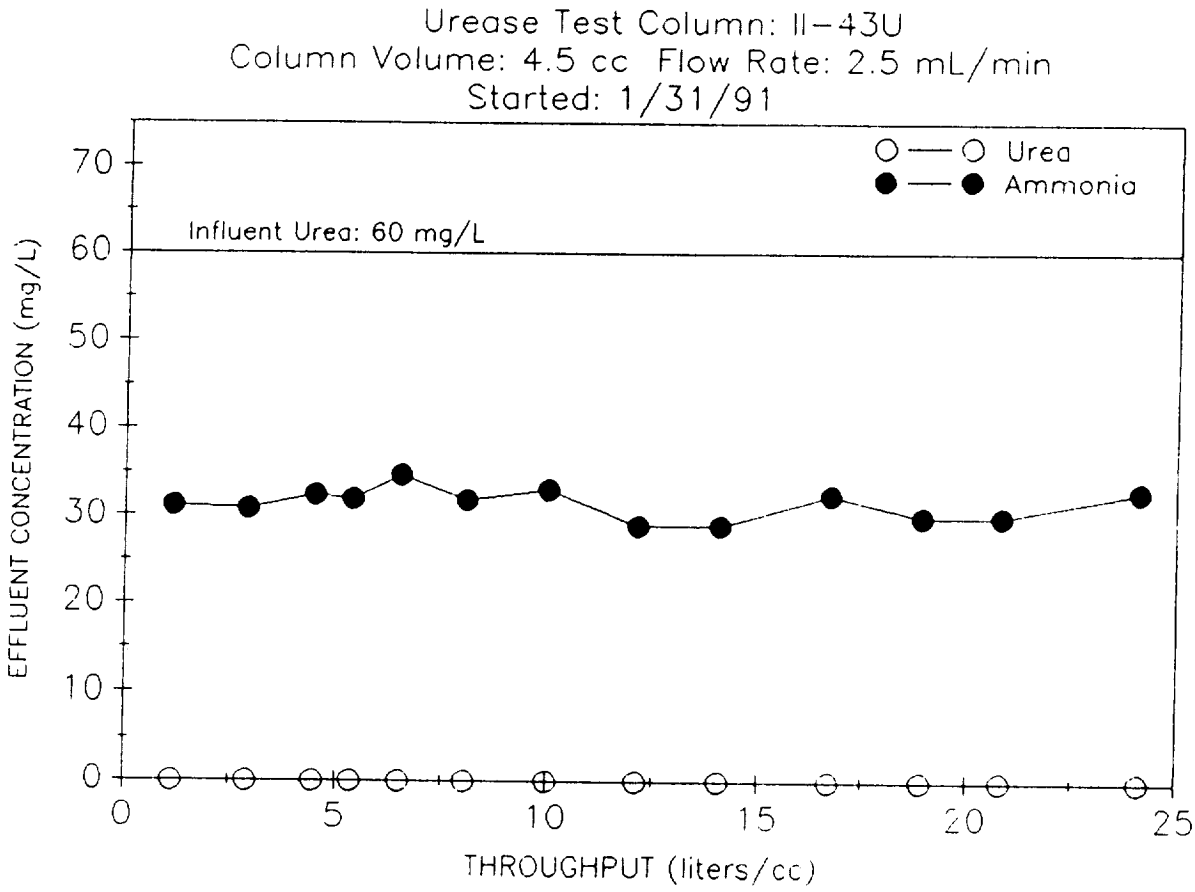

Urease Test Column: II-17UB
Column Volume: 7.4 cc Flow Rate: 2.5 mL/min
Started: 10/20/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0
4
8
12
16
20
24
28
32
THROUGHPUT (liters/cc)

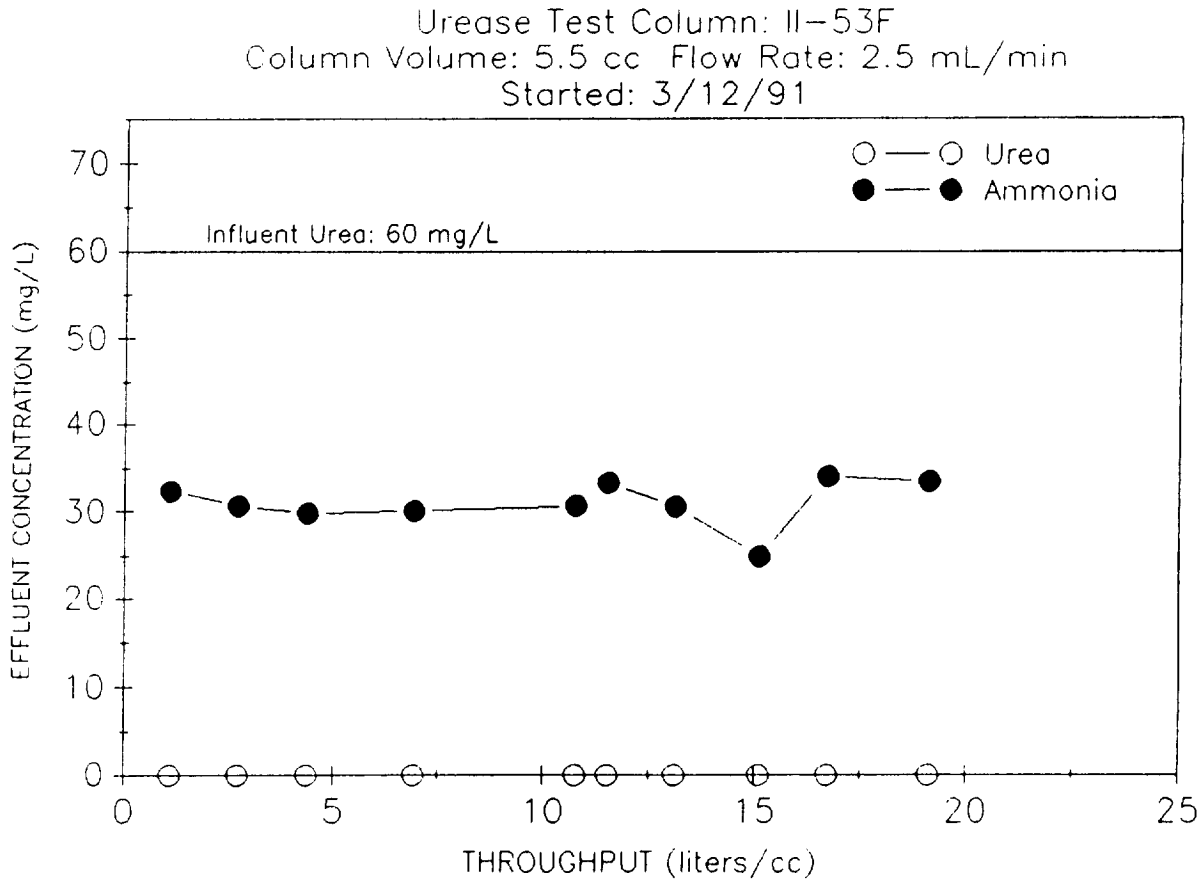

Urease Test Column: II-21U
Column Volume: 5.5 cc Flow Rate: 2.5 mL/min
Started: 11/27/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0
10
20
30
40
50
THROUGHPUT (liters/cc)

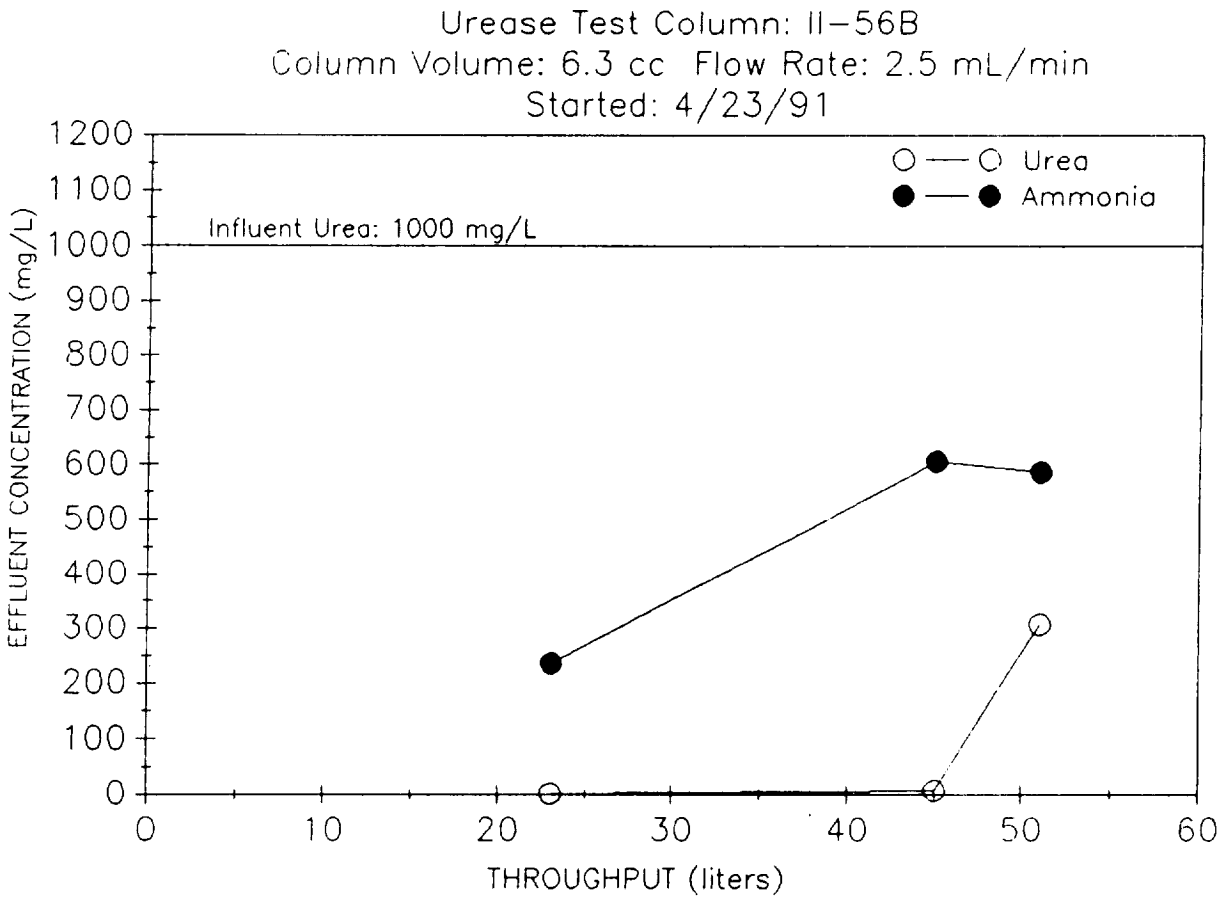

Urease Test Column: II–36UI
Column Volume: 5.5 cc Flow Rate: 2.0 mL/min
Started: 12/24/90
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0
4
8
12
16
20
THROUGHPUT (liters/cc)

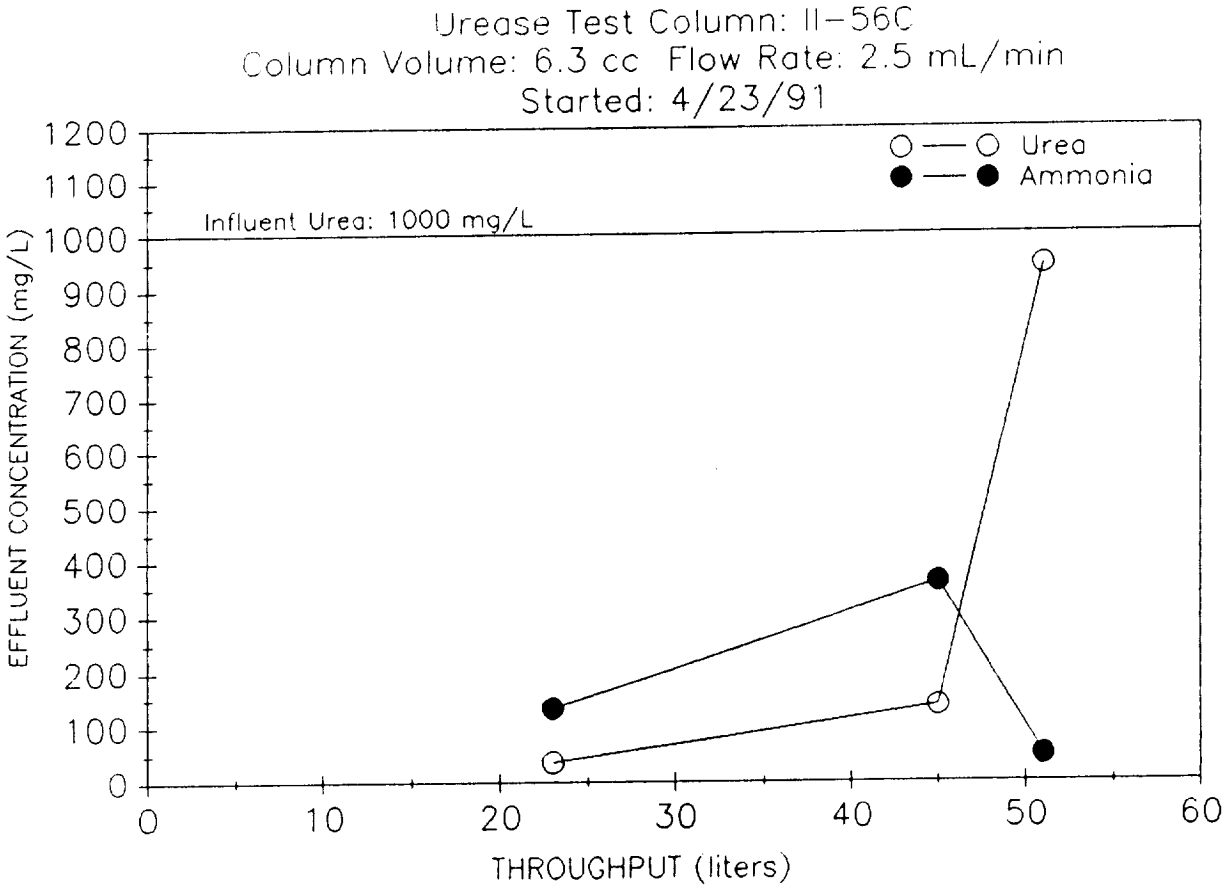

Urease Test Column: II-39A with 100 cc IRN-77
Column Volume: 5.5 cc Flow Rate: 2.2 mL/min
Started: 1/7/91
Urea
Ammonia
Post Ammonia
Influent Urea: 60 mg/L
20 ppm EtOH
Added at 18 L/cc
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters/cc)
0
10
20
30
40
50
60
70
0
5
10
15
20
25
30

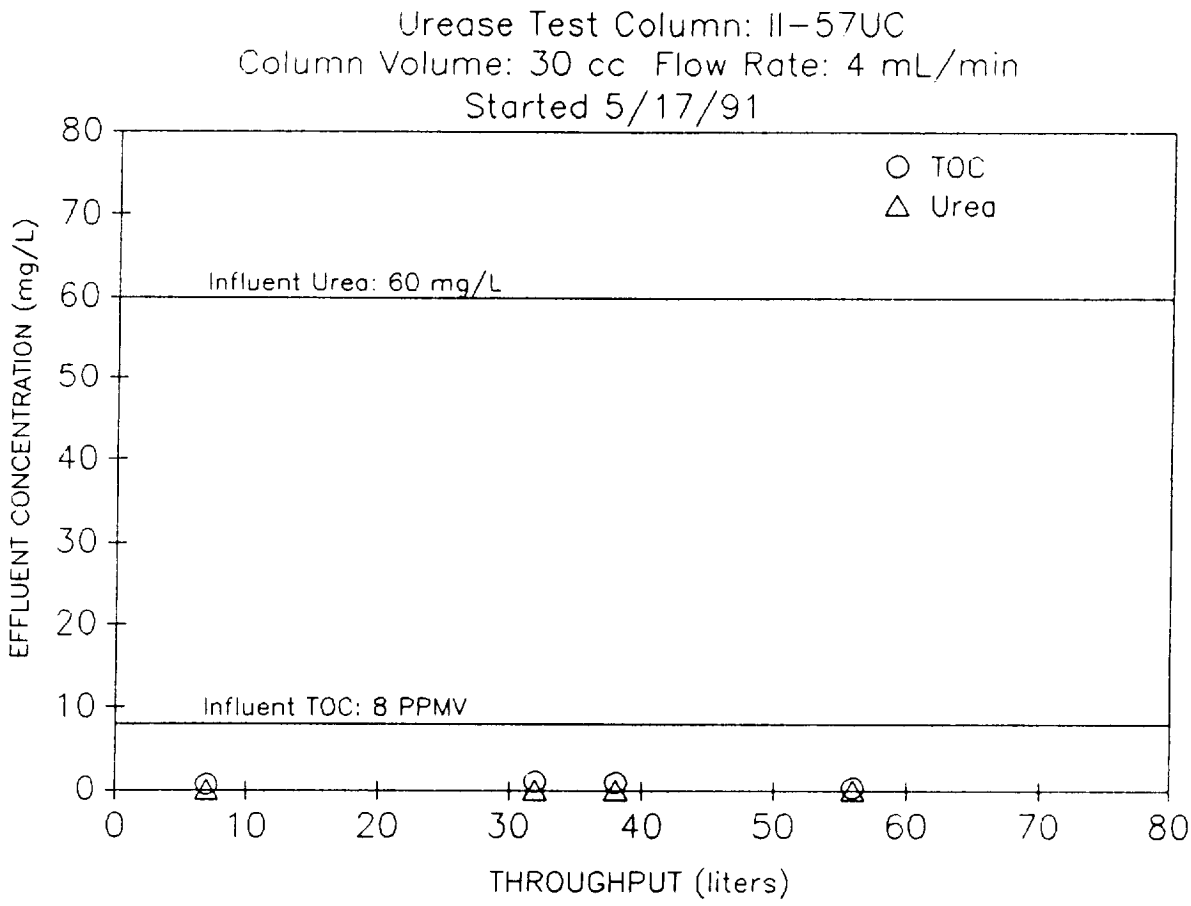

Urease Test Column: II-39C
Column Volume: 6.9 cc Flow Rate: 2.5 mL/min
Started: 3/5/91
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0
5
10
15
20
25
THROUGHPUT (liters/cc)

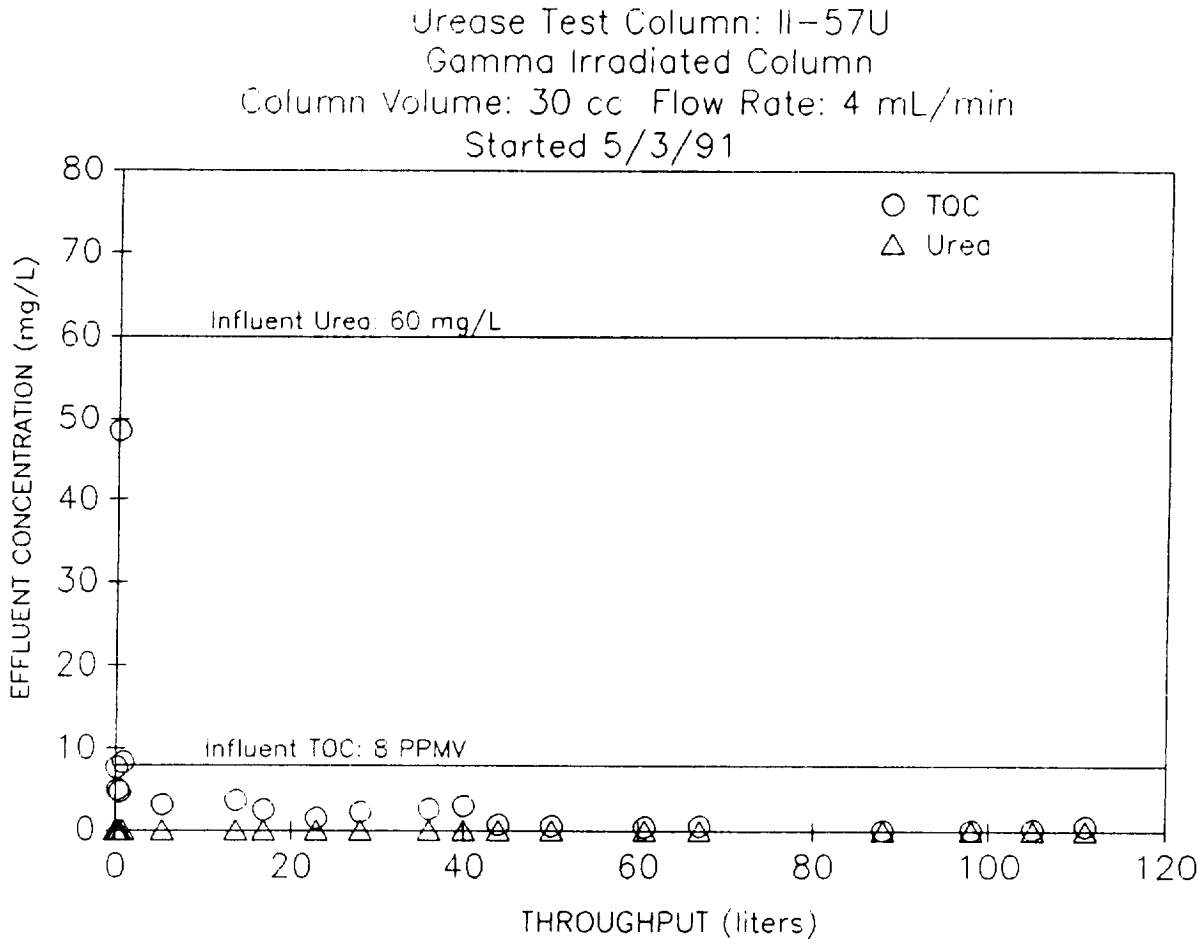

Urease Test Column: II-43U
Column Volume: 4.5 cc Flow Rate: 2.5 mL/min
Started: 1/31/91
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0
5
10
15
20
25
THROUGHPUT (liters/cc)


Urease Test Column: II-53F
Column Volume: 5.5 cc Flow Rate: 2.5 mL/min
Started: 3/12/91
Urea
Ammonia
Influent Urea: 60 mg/L
EFFLUENT CONCENTRATION (mg/L)
70
60
50
40
30
20
10
0
0
5
10
15
20
25
THROUGHPUT (liters/cc)


Urease Test Column: II-56B
Column Volume: 6.3 cc Flow Rate: 2.5 mL/min
Started: 4/23/91
Urea
Ammonia
Influent Urea: 1000 mg/L
EFFLUENT CONCENTRATION (mg/L)
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
0
10
20
30
40
50
60
THROUGHPUT (liters)


Urease Test Column: II-56C
Column Volume: 6.3 cc Flow Rate: 2.5 mL/min
Started: 4/23/91
Urea
Ammonia
Influent Urea: 1000 mg/L
EFFLUENT CONCENTRATION (mg/L)
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
0
10
20
30
40
50
60
THROUGHPUT (liters)


Urease Test Column: II-57UC
Column Volume: 30 cc Flow Rate: 4 mL/min
Started 5/17/91
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters)
TOC
Urea
Influent Urea: 60 mg/L
Influent TOC: 8 PPMV
0
10
20
30
40
50
60
70
80


Urease Test Column: II-57U
Gamma Irradiated Column
Column Volume: 30 cc Flow Rate: 4 mL/min
Started 5/3/91
EFFLUENT CONCENTRATION (mg/L)
THROUGHPUT (liters)
TOC
Urea
Influent Urea: 60 mg/L
Influent TOC: 8 PPMV
0
10
20
30
40
50
60
70
80
0
20
40
60
80
100
120